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Protection of
Environment

40

PARTS 61 TO 80
Revised as of July 1, 1990



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PARTS 61 TO 80

Revised as of July 1, 1990

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AS OF JULY 1, 1990

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Protection of
Environment

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Cite this Code: CFR

***To cite the regulations in
this volume use title,
part and section
number. Thus, 40 CFR
61.01 refers to title 40,
part 61, section 01.***

Explanation

The Code of Federal Regulations is a codification of the general and permanent rules published in the Federal Register by the Executive departments and agencies of the Federal Government. The Code is divided into 50 titles which represent broad areas subject to Federal regulation. Each title is divided into chapters which usually bear the name of the issuing agency. Each chapter is further subdivided into parts covering specific regulatory areas.

ISSUE DATES

Each volume of the Code is revised at least once each calendar year and issued on a quarterly basis approximately as follows:

Title 1 through Title 16.....	as of January 1
Title 17 through Title 27.....	as of April 1
Title 28 through Title 41.....	as of July 1
Title 42 through Title 50.....	as of October 1

The appropriate revision date is printed on the cover of each volume.

LEGAL STATUS

The contents of the Federal Register are required to be judicially noticed (44 U.S.C. 1507). The Code of Federal Regulations is prima facie evidence of the text of the original documents (44 U.S.C. 1510).

HOW TO USE THE CODE OF FEDERAL REGULATIONS

The Code of Federal Regulations is kept up to date by the individual issues of the Federal Register. These two publications must be used together to determine the latest version of any given rule.

To determine whether a Code volume has been amended since its revision date (in this case, July 1, 1990), consult the "List of CFR Sections Affected (LSA)," which is issued monthly, and the "Cumulative List of Parts Affected," which appears in the Reader Aids section of the daily Federal Register. These two lists will identify the Federal Register page number of the latest amendment of any given rule.

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Each volume of the Code contains amendments published in the Federal Register since the last revision of that volume of the Code. Source citations for the regulations are referred to by volume number and page number of the Federal Register and date of publication. Publication dates and effective dates are usually not the same and care must be exercised by the user in determining the actual effective date. In instances where the effective date is beyond the cut-off date for the Code a note has been inserted to reflect the future effective date. In those instances where a regulation published in the Federal Register states a

date certain for expiration, an appropriate note will be inserted following the text.

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The Paperwork Reduction Act of 1980 (Pub. L. 96-511) requires Federal agencies to display an OMB control number with their information collection request. Many agencies have begun publishing numerous OMB control numbers as amendments to existing regulations in the CFR. These OMB numbers are placed as close as possible to the applicable recordkeeping or reporting requirements.

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Provisions that become obsolete before the revision date stated on the cover of each volume are not carried. Code users may find the text of provisions in effect on a given date in the past by using the appropriate numerical list of sections affected. For the period before January 1, 1973, consult either the List of CFR Sections Affected, 1949-1963, or 1964-1972, published in three separate volumes. For the period beginning January 1, 1973, a "List of CFR Sections Affected" is published at the end of each CFR volume.

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What is incorporation by reference? Incorporation by reference was established by statute and allows Federal agencies to meet the requirement to publish regulations in the Federal Register by referring to materials already published elsewhere. For an incorporation to be valid, the Director of the Federal Register must approve it. The legal effect of incorporation by reference is that the material is treated as if it were published in full in the Federal Register (5 U.S.C. 552(a)). This material, like any other properly issued regulation, has the force of law.

What is a proper incorporation by reference? The Director of the Federal Register will approve an incorporation by reference only when the requirements of 1 CFR Part 51 are met. Some of the elements on which approval is based are:

- (a) The incorporation will substantially reduce the volume of material published in the Federal Register.
- (b) The matter incorporated is in fact available to the extent necessary to afford fairness and uniformity in the administrative process.
- (c) The incorporating document is drafted and submitted for publication in accordance with 1 CFR Part 51.

Properly approved incorporations by reference in this volume are listed in the Finding Aids at the end of this volume.

What if the material incorporated by reference cannot be found? If you have any problem locating or obtaining a copy of material listed in the Finding Aids of this volume as an approved incorporation by reference, please contact the agency that issued the regulation containing that incorporation. If, after contacting the agency, you find the material is not available, please notify the Director of the Federal Register, National Archives and Records Administration, Washington DC 20408, or call (202) 523-4534.

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A subject index to the Code of Federal Regulations is contained in a separate volume, revised annually as of January 1, entitled **CFR INDEX AND FINDING AIDS**. This volume contains the Parallel Table of Statutory Authorities and Agency Rules (Table I), and Acts Requiring Publication in the Federal Register (Table

III. A list of CFR Titles, Chapters, and Parts and an alphabetical list of agencies publishing in the CFR are also included in this volume.

An index to the text of "Title 3—The President" is carried within that volume.

The Federal Register Index is issued monthly in cumulative form. This index is based on a consolidation of the "Contents" entries in the daily Federal Register.

A List of CFR Sections Affected (LSA) is published monthly, keyed to the revision dates of the 50 CFR titles.

REPUBLICATION OF MATERIAL

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MARTHA L. GIRARD,

Director,

Office of the Federal Register.

July 2, 1990.

THIS TITLE

Title 40—PROTECTION OF ENVIRONMENT is composed of fifteen volumes. The parts in these volumes are arranged in the following order: Parts 1-51, Part 52, Parts 53-60, Parts 61-80, Parts 81-85, Parts 86-99, Parts 100-149, Parts 150-189, Parts 190-259, Parts 260-299, Parts 300-399, Parts 400-424, Parts 425-699, Parts 700-789 and Part 790 to End. A derivation table appears in the volume containing Parts 1-51. The contents of these volumes represent all current regulations codified under this title of the CFR as of July 1, 1990.

Chapter I—Environmental Protection Agency appears in all fifteen volumes. A Pesticide Tolerance Commodity/Chemical Index appears in Parts 150-189. A Toxic Substances Chemical—CAS Number Index appears in Parts 700-789 and Part 790 to End. Distribution Tables appear in the volumes containing Parts 100-149, Parts 190-259 and Parts 260-299. Redesignation Tables appear in the volumes containing Parts 1-51, Parts 81-85, Parts 86-99, Parts 150-189, and Parts 700-789. Regulations issued by the Council on Environmental Quality appear in the volume containing Part 790 to End.

For this volume, Sheli Fleming was Chief Editor. The Code of Federal Regulations publication program is under the direction of Richard L. Claypoole, assisted by Alomha S. Morris.

if any changes have been made to the Code of Federal Regulations or what documents have been published in the Federal Register without reading the Federal Register every day? If so, you may wish to subscribe to the LSA (*List of CFR Sections Affected*), the *Federal Register Index*, or both.

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APPENDIX A—NATIONAL EMISSION STANDARDS FOR HAZARDOUS AIR POLLUTANTS, COMPLIANCE STATUS INFORMATION

APPENDIX B—TEST METHODS

APPENDIX C—QUALITY ASSURANCE PROCEDURES

AUTHORITY: Secs. 101, 112, 114, 116, 301, Clean Air Act as amended (42 U.S.C. 7401, 7412, 7414, 7416, 7601).

SOURCE: 38 FR 8826, Apr. 6, 1973, unless otherwise noted.

Subpart A—General Provisions

§ 61.01 Lists of pollutants and applicability of part 61.

(a) The following list presents the substances that, pursuant to section 112 of the Act, have been designated as hazardous air pollutants. The FEDERAL REGISTER citations and dates refer to the publication in which the listing decision was originally published.

- Asbestos (36 FR 5931; Mar. 31, 1971)
- Benzene (42 FR 29332; June 8, 1977)
- Beryllium (36 FR 5931; Mar. 31, 1971)
- Coke Oven Emissions (49 FR 36560; Sept. 18, 1984)
- Inorganic Arsenic (45 FR 37886; June 5, 1980)
- Mercury (36 FR 5931; Mar. 31, 1971)
- Radionuclides (44 FR 76738; Dec. 27, 1979)
- Vinyl Chloride (40 FR 59532; Dec. 24, 1975)

(b) The following list presents other substances for which a FEDERAL REGISTER notice has been published that included consideration of the serious health effects, including cancer, from ambient air exposure to the substance.

- Acrylonitrile (50 FR 24319; June 10, 1985)
- 1,3-Butadiene (50 FR 41466; Oct. 10, 1985)
- Cadmium (50 FR 42000; Oct. 16, 1985)

- Carbon Tetrachloride (50 FR 32621; Aug. 13, 1985)
- Chlorinated Benzenes (50 FR 32628; Aug. 13, 1985)
- Chlorofluorocarbon—113 (50 FR 24313; June 10, 1985)
- Chloroform (50 FR 39626; Sept. 27, 1985)
- Chloroprene (50 FR 39632; Sept. 27, 1985)
- Chromium (50 FR 24317; June 10, 1985)
- Copper (52 FR 5496; Feb. 23, 1987)
- Epichlorohydrin (50 FR 24575; June 11, 1985)
- Ethylene Dichloride (50 FR 41994; Oct. 16, 1985)
- Ethylene Oxide (50 FR 40286; Oct. 2, 1985)
- Hexachlorocyclopentadiene (50 FR 40154; Oct. 1, 1985)
- Manganese (50 FR 32627; Aug. 13, 1985)
- Methyl Chloroform (50 FR 24314; June 10, 1985)
- Methylene Chloride (50 FR 42037; Oct. 17, 1985)
- Nickel (51 FR 34135; Sept. 25, 1986)
- Perchloroethylene (50 FR 52800; Dec. 26, 1985)
- Phenol (51 FR 22854; June 23, 1986)
- Polycyclic Organic Matter (49 FR 31680; Aug. 8, 1984)
- Toluene (49 FR 22195; May 25, 1984)
- Trichloroethylene (50 FR 52422; Dec. 23, 1985)
- Vinylidene Chloride (50 FR 32632; Aug. 13, 1985)
- Zinc and Zinc Oxide (52 FR 32597; Aug. 28, 1987)

(c) This part applies to the owner or operator of any stationary source for which a standard is prescribed under this part.

[50 FR 46290, Nov. 7, 1985, as amended at 51 FR 7715 and 7719, Mar. 5, 1986; 51 FR 11022, Apr. 1, 1986; 52 FR 37617, Oct. 8, 1987]

§ 61.02 Definitions.

The terms used in this part are defined in the Act or in this section as follows:

Act means the Clean Air Act (42 U.S.C. 7401 et seq.).

Administrator means the Administrator of the Environmental Protection Agency or his authorized representative.

Alternative method means any method of sampling and analyzing for an air pollutant which is not a reference method but which has been demonstrated to the Administrator's satisfaction to produce results adequate for the Administrator's determination of compliance.

Capital expenditure means an expenditure for a physical or operational change to a stationary source which exceeds the product of the applicable "annual asset guideline repair allowance percentage" specified in the latest edition of Internal Revenue Service (IRS) Publication 534 and the stationary source's basis, as defined by section 1012 of the Internal Revenue Code. However, the total expenditure for a physical or operational change to a stationary source must not be reduced by any "excluded additions" as defined for stationary sources constructed after December 31, 1981, in IRS Publication 534, as would be done for tax purposes. In addition, "annual asset guideline repair allowance" may be used even though it is excluded for tax purposes in IRS Publication 534.

Commenced means, with respect to the definition of "new source" in section 111(a)(2) of the Act, that an owner or operator has undertaken a continuous program of construction or modification or that an owner or operator has entered into a contractual obligation to undertake and complete, within a reasonable time, a continuous program of construction or modification.

Compliance schedule means the date or dates by which a source or category of sources is required to comply with the standards of this part and with any steps toward such compliance which are set forth in a waiver of compliance under § 61.11.

Construction means fabrication, erection, or installation of an affected facility.

Effective date is the date of promulgation in the FEDERAL REGISTER of an applicable standard or other regulation under this part.

Existing source means any stationary source which is not a new source.

Monitoring system means any system, required under the monitoring sections in applicable subparts, used to sample and condition (if applicable), to analyze, and to provide a record of emissions or process parameters.

New source means any stationary source, the construction or modification of which is commenced after the publication in the FEDERAL REGISTER of proposed national emission stand-

ards for hazardous air pollutants which will be applicable to such source.

Owner or operator means any person who owns, leases, operates, controls, or supervises a stationary source.

Reference method means any method of sampling and analyzing for an air pollutant, as described in Appendix B to this part.

Run means the net period of time during which an emission sample is collected. Unless otherwise specified, a run may be either intermittent or continuous within the limits of good engineering practice.

Standard means a national emission standard including a design, equipment, work practice or operational standard for a hazardous air pollutant proposed or promulgated under this part.

Startup means the setting in operation of a stationary source for any purpose.

Stationary source means any building, structure, facility, or installation which emits or may emit any air pollutant which has been designated as hazardous by the Administrator.

[44 FR 55174, Sept. 25, 1979, as amended at 50 FR 46290, Nov. 7, 1985]

§ 61.03 Units and abbreviations.

Used in this part are abbreviations and symbols of units of measure. These are defined as follows:

(a) System International (SI) units of measure:

A = ampere
g = gram
Hz = hertz
J = joule
K = degree Kelvin
kg = kilogram
m = meter
m² = square meter
m³ = cubic meter
mg = milligram = 10⁻³ gram
mm = millimeter = 10⁻³ meter
Mg = megagram = 10⁶ gram
mol = mole
N = newton
ng = nanogram = 10⁻⁹ gram
nm = nanometer = 10⁻⁹ meter
Pa = pascal
s = second
V = volt
W = watt
Ω = ohm

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μg = microgram = 10^{-6} gram

(b) Other units of measure:

$^{\circ}\text{C}$ = degree Celsius (centigrade)
 cfm = cubic feet per minute
 cc = cubic centimeter
 Ci = curie
 d = day
 $^{\circ}\text{F}$ = degree Fahrenheit
 ft^2 = square feet
 ft^3 = cubic feet
 gal = gallon
 in = inch
 in Hg = inches of mercury
 $\text{in H}_2\text{O}$ = inches of water
 l = liter
 lb = pound
 lpn = liter per minute
 min = minute
 ml = milliliter = 10^{-3} liter
 mrem = millirem = 10^{-3} rem
 oz = ounces
 pCi = picocurie = 10^{-12} curie
 psig = pounds per square inch gage
 $^{\circ}\text{R}$ = degree Rankine
 μl = microliter = 10^{-6} liter
 v/v = volume per volume
 yd^2 = square yards
 yr = year

(c) Chemical nomenclature:

Be = beryllium
 Hg = mercury
 H_2O = water

(d) Miscellaneous:

act = actual
 avg = average
 I.D. = inside diameter
 M = molar
 N = normal
 O.D. = outside diameter
 $\%$ = percent
 std = standard

[42 FR 51574, Sept. 29, 1977, as amended at 54 FR 51704, Dec. 15, 1989]

\$ 61.04 Address.

(a) All requests, reports, applications, submittals, and other communications to the Administrator pursuant to this part shall be submitted in duplicate to the appropriate Regional Office of the U.S. Environmental Protection Agency to the attention of the Director of the Division indicated in the following list of EPA Regional Offices.

Region I (Connecticut, Maine, Massachusetts, New Hampshire, Rhode Island, Vermont), Director, Air Management Division, U.S. Environmental Protection Agency, John F. Kennedy Federal Building, Boston, MA 02203.

Region II (New Jersey, New York, Puerto Rico, Virgin Islands), Director, Air and Waste Management Division, U.S. Environmental Protection Agency, Federal Office Building, 26 Federal Plaza (Foley Square), New York, NY 10278.

Region III (Delaware, District of Columbia, Maryland, Pennsylvania, Virginia, West Virginia), Director, Air and Waste Management Division, U.S. Environmental Protection Agency, Curtis Building, Sixth and Walnut Streets, Philadelphia, PA 19106.

Region IV (Alabama, Florida, Georgia, Kentucky, Mississippi, North Carolina, South Carolina, Tennessee), Director, Air and Waste Management Division, U.S. Environmental Protection Agency, 345 Courtland Street, NE, Atlanta, GA 30365.

Region V (Illinois, Indiana, Michigan, Minnesota, Ohio, Wisconsin), Director, Air Management Division, U.S. Environmental Protection Agency, 230 South Dearborn Street, Chicago, IL 60604.

Region VI (Arkansas, Louisiana, New Mexico, Oklahoma, Texas), Director, Air, Pesticides, and Toxics Division, U.S. Environmental Protection Agency, 1445 Ross Avenue, Dallas, Texas 75202.

Region VII (Iowa, Kansas, Missouri, Nebraska), Director, Air and Toxics Division, U.S. Environmental Protection Agency, 726 Minnesota Avenue, Kansas City, KS 66101.

Region VIII (Colorado, Montana, North Dakota, South Dakota, Utah, Wyoming), Director, Air and Waste Management Division, U.S. Environmental Protection Agency, 1860 Lincoln Street, Denver, CO 80295.

Region IX (American Samoa, Arizona, California, Guam, Hawaii, Nevada), Director, Air and Waste Management Division, U.S. Environmental Protection Agency, 215 Fremont Street, San Francisco, CA 94105.

Region X (Alaska, Oregon, Idaho, Washington), Director, Air and Waste Management Division, U.S. Environmental Protection Agency, 1200 Sixth Avenue, Seattle, WA 98101.

(b) Section 112(d) directs the Administrator to delegate to each State, when appropriate, the authority to implement and enforce national emission standards for hazardous air pollutants for stationary sources located in such State. If the authority to implement and enforce a standard under this part has been delegated to a State, all information required to be submitted to EPA under paragraph (a) of this section shall also be submitted to the appropriate State agency (provided, that each specific delegation may exempt

sources from a certain Federal or State reporting requirement). The Administrator may permit all or some of the information to be submitted to the appropriate State agency only, instead of to EPA and the State agency. The appropriate mailing address for those States whose delegation request has been approved is as follows:

(A) [Reserved]

(B) State of Alabama, Air Pollution Control Division, Air Pollution Control Commission, 645 S. McDonough Street, Montgomery, AL 36104.

(C) State of Alaska, Department of Environmental Conservation, 3220 Hospital Drive, Juneau, AK 99811.

(D) Arizona.

Arizona Department of Health Services, 1740 West Adams Street, Phoenix, AZ 85007.

Maricopa County Department of Health Services, Bureau of Air Pollution Control, 1825 East Roosevelt Street, Phoenix, AZ 85006.

Pima County Health Department, Air Quality Control District, 151 West Congress, Tucson, AZ 85701.

(E) State of Arkansas: Chief, Division of Air Pollution Control, Arkansas Department of Pollution Control and Ecology, 8001 National Drive, P.O. Box 9583, Little Rock, Arkansas 72209.

(F) California.

Amador County Air Pollution Control District, P.O. Box 430, 810 Court Street, Jackson, CA 95642.

Bay Area Air Pollution Control District, 939 Ellis Street, San Francisco, CA 94109.

Butte County Air Pollution Control District, P.O. Box 1229, 316 Nelson Avenue, Oroville, CA 95965.

Calaveras County Air Pollution Control District, Government Center, El Dorado Road, San Andreas, CA 95249.

Colusa County Air Pollution Control District, 751 Fremont Street, Colusa, CA 95952.

El Dorado Air Pollution Control District, 330 Fair Lane, Placerville, CA 95667.

Fresno County Air Pollution Control District, 1221 Fulton Mall, Fresno, CA 93721.

Glenn County Air Pollution Control District, P.O. Box 351, 720 North Colusa Street, Willows, CA 95988.

Great Basin Unified Air Pollution Control District, 157 Short Street, Suite 6, Bishop, CA 93514.

Imperial County Air Pollution Control District, County Services Building, 939 West Main Street, El Centro, CA 92243.

Kern County Air Pollution Control District, 1601 H Street, Suite 250, Bakersfield, CA 93301.

Kings County Air Pollution Control District, 330 Campus Drive, Hanford, CA 93230.

Lake County Air Pollution Control District, 255 North Forbes Street, Lakeport, CA 95453.

Lassen County Air Pollution Control District, 175 Russell Avenue, Susanville, CA 96130.

Madera County Air Pollution Control District, 135 West Yosemite Avenue, Madera, CA 93637.

Mariposa County Air Pollution Control District, Box 5, Mariposa, CA 95338.

Mendocino County Air Pollution Control District, County Courthouse, Ukiah, CA 94582.

Merced County Air Pollution Control District, P.O. Box 471, 240 East 15th Street, Merced, CA 95340.

Modoc County Air Pollution Control District, 202 West 4th Street, Alturas, CA 96101.

Monterey Bay Unified Air Pollution Control, 1164 Monroe Street, Suite 10, Salinas, CA 93906.

Nevada County Air Pollution Control District, H.E.W. Complex, Nevada City, CA 95959.

North Coast Unified Air Quality Management District, 5630 South Broadway, Eureka CA 95501.

Northern Sonoma County Air Pollution Control District, 134 "A" Avenue, Auburn, CA 95448.

Placer County Air Pollution Control District, 11491 "B" Avenue, Auburn, CA 95603.

Plumas County Air Pollution Control District, P.O. Box 480, Quincy, CA 95971.

Sacramento County Air Pollution Control District, 3701 Branch Center Road, Sacramento, CA 95827.

San Bernardino County Air Pollution Control District, 15579-8th, Victorville, CA 92392.

San Diego County Air Pollution Control District, 9150 Chesapeake Drive, San Diego, CA 92123.

San Joaquin County Air Pollution Control District, 1601 East Hazelton Street (P.O. Box 2009), Stockton, CA 95201.

San Luis Obispo County Air Pollution Control District, P.O. Box 637, San Luis Obispo, CA 93406.

Santa Barbara County Air Pollution Control District, 315 Camino del Rimedio, Santa Barbara, CA 93110.

Shasta County Air Pollution Control District, 2650 Hospital Lane, Redding, CA 96001.

Sierra County Air Pollution Control District, P.O. Box 286, Downieville, CA 95936.

Siskiyou County Air Pollution Control District, 525 South Foothill Drive, Yreka, CA 96097.

South Coast Air Quality Management District, 9150 Flair Drive, El Monte, CA 91731.

Stanislaus County Air Pollution Control District, 1030 Scenic Drive, Modesto, CA 95350.

Sutter County Air Pollution Control District, Sutter County Office Building, 142 Garden Highway, Yuba City, CA 95991.

Tehama County Air Pollution Control District, P.O. Box 38, 1760 Walnut Street, Red Bluff, CA 96080.

Tulare County Air Pollution Control District, County Civic Center, Visalia, CA 93277.

Tuolumne County Air Pollution Control District, 9 North Washington Street, Sonoma, CA 95370.

Ventura County Air Pollution Control District, 800 South Victoria Avenue, Ventura, CA 93009.

Yolo-Solano Air Pollution Control District, P.O. Box 1006, 323 First Street, S.W., Woodland, CA 95695.

(G) State of Colorado, Department of Health, Air Pollution Control Division, 4210 East 11th Avenue, Denver, CO 80220.

EDITORIAL NOTE: For a table listing Region VIII's NESHAPs delegation status, see paragraph (c) of this section.

(H) State of Connecticut, Department of Environmental Protection, State Office Building, Hartford, CN 06115.

(I) State of Delaware:
Delaware Department of Natural Resources and Environmental Control, Tatnall Building, P.O. Box 1401, Dover, DE 19901.

(J) [Reserved]
(K) Bureau of Air Quality Management, Department of Environmental Regulation, Twin Towers Office Building, 2600 Blair Stone Road, Tallahassee, FL 32301.

(L) State of Georgia, Environmental Protection Division, Department of Natural Resources, 270 Washington Street, S.W., Atlanta, GA 30334.

(M) Hawaii Department of Health, 1250 Punchbowl Street, Honolulu, HI 96813.

Hawaii Department of Health (mailing address), Post Office Box 3378, Honolulu, HI 96801.

(N)-(O) [Reserved]

(P) State of Indiana, Indiana Department of Environmental Management, 105 South Meridian Street, P.O. Box 6015, Indianapolis, Indiana 46206.

(Q) State of Iowa: Iowa Department of Natural Resources, Environmental Protection Division, Henry A. Wallace Building, 900 East Grand, Des Moines, IA 50319.

(R) State of Kansas: Kansas Department of Health and Environment, Bureau of Air Quality and Radiation Control, Forbes Field, Topeka, KS 66620.

(S) Division of Air Pollution Control, Department for Natural Resources and Envi-

ronmental Protection, U.S. 127, Frankfort, KY 40601.

(T) State of Louisiana: Program Administrator, Air Quality Division, Louisiana Department of Environmental Quality, P.O. Box 44096, Baton Rouge, Louisiana 70804.

(U) State of Maine, Department of Environmental Protection, State House, Augusta, ME 04330.

(V) State of Maryland, Bureau of Air Quality and Noise Control, Maryland State Department of Health and Mental Hygiene, 201 West Preston Street, Baltimore, MD 21201.

(W) Commonwealth of Massachusetts: Massachusetts Department of Environmental Quality Engineering, Division of Air Quality Control, One Winter Street, Boston, MA 02108.

(X) State of Michigan, Air Pollution Control Division, Michigan Department of Natural Resources, Stevens T. Mason Building, 8th Floor, Lansing, MI 48926.

(Y) Minnesota Pollution Control Agency, Division of Air Quality, 520 Lafayette Road, St. Paul, Minnesota 55155.

(Z) Bureau of Pollution Control, Department of Natural Resources, P.O. Box 10385, Jackson, MS 39209.

(AA) State of Missouri: Missouri Department of Natural Resources, Division of Environmental Quality, P.O. Box 176, Jefferson City, MO 65102.

(BB) State of Montana, Department of Health and Environmental Services, Air Quality Bureau, Cogswell Building, Helena, MT 59601.

EDITORIAL NOTE: For a table listing Region VIII's NESHAPs delegation status, see paragraph (c) of this section.

(CC) State of Nebraska, Nebraska Department of Environmental Control, P.O. Box 94877, State House Station, Lincoln, NB 68509.

Lincoln-Lancaster County Health Department, Division of Environmental Health, 2200 St. Marys Avenue, Lincoln, NB 68502.

(DD) Nevada.
Clark County, County District Health Department, Air Pollution Control Division, 625 Shadow Lane, Las Vegas, NV 89106.

Nevada Department of Conservation and Natural Resources, Division of Environmental Protection, 201 South Fall Street, Carson City, NV 89710.

Washoe County District Health Department, Division of Environmental Protection, 10 Kirman Avenue, Reno, NV 89502.

(EE) State of New Hampshire: New Hampshire Air Resources Agency, Health and Welfare Building, Hazen Drive, Concord, NH 03301.

(FF) State of New Jersey: New Jersey Department of Environmental Protection, John Fitch Plaza, P.O. Box 2807, Trenton, NJ 08625.

(GG) State of New Mexico: Director, New Mexico Environmental Improvement Division, Health and Environment Department, 1190 St. Francis Drive, Santa Fe, New Mexico 87503.

(I) The City of Albuquerque and Bernalillo County: Director, The Albuquerque Environmental Health Department, The City of Albuquerque, P.O. Box 1293, Albuquerque, New Mexico 87103.

(HH) New York: New York State Department of Environmental Conservation, 50 Wolf Road, Albany, NY 12233, attention: Division of Air Resources.

(II) North Carolina Environmental Management Commission, Department of Natural and Economic Resources, Division of Environmental Management, P.O. Box 27687, Raleigh, NC 27611. Attention: Air Quality Section.

(JJ) State of North Dakota, State Department of Health and Consolidated Laboratories, Division of Environmental Engineering, State Capitol, Bismarck, ND 58505.

EDITORIAL NOTE: For a table listing Region VIII's NESHAPs delegation status, see paragraph (c) of this section.

(KK) State of Ohio—

(i) Medina, Summit and Portage Counties: Director, Akron Regional Air Quality Management District, 177 South Broadway, Akron, OH 44308.

(ii) Stark County: Director, Air Pollution Control Division, Canton City Health Department, City Hall Annex Second Floor, 218 Cleveland Avenue S.W., Canton, OH 44702.

(iii) Butler, Clermont, Hamilton and Warren Counties: Director, Southwestern Ohio Air Pollution Control Agency, 2400 Beekman Street, Cincinnati, OH 45214.

(iv) Cuyahoga County: Commissioner, Division of Air Pollution Control, Department of Public Health and Welfare, 2735 Broadway Avenue, Cleveland, OH 44115.

(v) Belmont, Carroll, Columbiana, Harrison, Jefferson, and Monroe Counties: Director, North Ohio Valley Air Authority (NOVAA), 814 Adams Street, Steubenville, OH 43952.

(vi) Clark, Darke, Greene, Miami, Montgomery, and Preble Counties: Supervisor, Regional Air Pollution Control Agency (RAPCA), Montgomery County Health Department, 451 West Third Street, Dayton, OH 45402.

(vii) Lucas County and the City of Rossford (in Wood County): Director, Toledo Environmental Services Agency, 26 Main Street, Toledo, OH 43605.

(viii) Adams, Brown, Lawrence, and Scioto Counties: Engineer-Director, Air Division,

Portsmouth City Health Department, 740 Second Street, Portsmouth, OH 45662.

(ix) Allen, Ashland, Auglaize, Crawford, Defiance, Erie, Fulton, Hancock, Hardin, Henry, Huron, Marion, Mercer, Ottawa, Paulding, Putnam, Richland, Sandusky, Seneca, Van West, Williams, Wood (except City of Rossford), and Wyandot Counties: Ohio Environmental Protection Agency, Northwest District Office, Air Pollution Unit, 1035 Dezlaz Grove Drive, Bowling Green, OH 43402.

(x) Ashtabula, Holmes, Lorain, and Wayne Counties: Ohio Environmental Protection Agency, Northeast District Office, Air Pollution Unit, 2110 East Aurora Road, Twinsburg, OH 44087.

(xi) Athens, Coshocton, Gallia, Guernsey, Hocking, Jackson, Meigs, Morgan, Muskingum, Noble, Perry, Pike, Ross, Tuscarawas, Vinton, and Washington Counties: Ohio Environmental Protection Agency, Southeast District Office, Air Pollution Unit, 2195 Front Street, Logan, OH 43138.

(xii) Champaign, Clinton, Highland, Logan, and Shelby Counties: Ohio Environmental Protection Agency, Southwest District Office, Air Pollution Unit, East Fourth Street, Dayton, OH 45402.

(xiii) Delaware, Fairfield, Fayette, Franklin, Knox, Licking, Madison, Morrow, Pickaway, and Union Counties: Ohio Environmental Protection Agency, Central District Office, Air Pollution Unit, P.O. Box 1049, Columbus, OH 43266-0149.

(xiv) Geauga and Lake Counties: Lake County General Health District, Air Pollution Control, 105 Main Street, Painesville, OH 44077.

(xv) Mahoning and Trumbull Counties: Mahoning-Trumbull Air Pollution Control Agency, 9 West Front Street, Youngstown, OH 44503.

(LL) State of Oklahoma, Oklahoma State Department of Health, Air Quality Service, P.O. Box 53551, Oklahoma City, OK 73152.

(i) Oklahoma City and County: Director, Oklahoma City-County Health Department, 921 Northeast 23rd Street, Oklahoma City, Oklahoma 73105.

(ii) Tulsa County: Tulsa City-County Health Department, 4616 East Fifteenth Street, Tulsa, OK 74112.

(MM) State of Oregon, Department of Environmental Quality, Yeon Building, 522 SW. Fifth, Portland, OR 97204.

(i)-(vii) [Reserved]

(viii) Lane Regional Air Pollution Authority, 225 North Fifth, Suite 501, Springfield, OR 97477.

(NN) Pennsylvania.

(i) City of Philadelphia: Philadelphia Department of Public Health, Air Management Services, 500 S. Broad Street, Philadelphia, PA 19146.

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(ii) Commonwealth of Pennsylvania: Department of Environmental Resources, Post Office Box 2063, Harrisburg, PA 17120.

(iii) Allegheny County: Allegheny County Health Department, Bureau of Air Pollution Control, 301 Thirty-ninth Street, Pittsburgh, PA, 15201.

(OO) State of Rhode Island: Rhode Island Department of Environmental Management, 204 Cannon Building, Davis Street, Providence, RI 02908.

(PP) State of South Carolina, Office of Environmental Quality Control, Department of Health and Environmental Control, 2600 Bull Street, Columbia, SC 29201.

(QQ) State of South Dakota, Department of Water and Natural Resources, Office of Air Quality and Solid Waste, Joe Foss Building, 523 East Capitol, Pierre, SD 57501-3181.

EDITORIAL NOTE: For a table listing Region VIII's NESHAPs delegation status, see paragraph (c) of this section.

(RR) Division of Air Pollution Control, Tennessee Department of Public Health, 256 Capitol Hill Building, Nashville, TN 37219.

Knox County Department of Air Pollution, City/County Building, Room L222, 400 Main Avenue, Knoxville, TN 37902.

Air Pollution Control Bureau, Metropolitan Health Department, 311 23rd Avenue North, Nashville, TN 37203.

(SS) State of Texas, Texas Air Control Board, 6330 Highway 290 East, Austin, TX 78723.

(TT) State of Utah, Department of Health, Bureau of Air Quality, 288 North 1460 West, P.O. Box 16690, Salt Lake City, UT 84116-0690.

EDITORIAL NOTE: For a table listing Region VIII's NESHAPs delegation status, see paragraph (c) of this section.

(UU) State of Vermont: Vermont Agency of Environmental Conservation, Air Pollu-

tion Control, State Office Building, Montpelier, VT 05602.

(VV) Commonwealth of Virginia, Virginia State Air Pollution Control Board, Room 1106, Ninth Street Office Building, Richmond, VA 23219.

(WW)(i) Washington; State of Washington, Department of Ecology, Olympia, WA 98504.

(ii) Northwest Air Pollution Authority, 207 Pioneer Building, Second and Pine Streets, Mount Vernon, WA 98273.

(iii) Puget Sound Air Pollution Control Agency, 200 West Mercer Street, Room 205, Seattle, WA 98119-3958.

(iv) Spokane County Air Pollution Control Authority, North 811 Jefferson, Spokane, WA 99201.

(v) Yakima County Clean Air Authority, County Courthouse, Yakima, WA 98901.

(vi) Olympic Air Pollution Control Authority, 120 East State Avenue, Olympia, WA 98501.

(vii) Southwest Air Pollution Control Authority, Suite 7601 H, NE Hazel Dell Avenue, Vancouver, WA 98665.

(viii) Grant County Clean Air Authority, P.O. Box 37, Ephrata, WA 98823.

(XX) State of West Virginia: Air Pollution Control Commission, 1558 Washington Street, East, Charleston, WV 25311.

(ZZ) Wisconsin—Wisconsin Department of Natural Resources, P.O. Box 7921, Madison, WI 53707.

(ZZ)-(AAA) (Reserved)

(BBB) Commonwealth of Puerto Rico: Commonwealth of Puerto Rico Environmental Quality Board, P.O. Box 11785, San-turce, PR 00910.

(CCC) U.S. Virgin Islands: U.S. Virgin Islands Department of Conservation and Cultural Affairs, P.O. Box 578, Charlotte Amalie, St. Thomas, U.S. Virgin Islands 00801.

(c) The following is a table indicating the delegation status of National Emission Standards for Hazardous Air Pollutants in Region VIII.

DELEGATION STATUS OF NATIONAL EMISSION STANDARD FOR HAZARDOUS AIR POLLUTANTS

[(NESHAPs) for Region VIII]

Subject	State					
	CO	MT	ND	SD	UT	WY
A General Provisions.....	(*)	(*)	(*)	(*)	(*)	
C Beryllium.....	(*)	(*)	(*)		(*)	
D Beryllium Rocket Motor Firing.....	(*)	(*)	(*)		(*)	
E Mercury.....	(*)	(*)	(*)		(*)	
F Vinyl Chloride.....	(*)	(*)	(*)		(*)	
J Equipment Leaks (Fugitive Emission Sources) of Benzene.....	(*)	(*)	(*)		(*)	
M Asbestos.....	(*)	(*)	(*)		(*)	
N Inorganic Arsenic Emissions from Glass Manufacturing Plants.....	(*)	(*)	(*)	(*)	(*)	(*) 1

**DELEGATION STATUS OF NATIONAL EMISSION STANDARD FOR HAZARDOUS AIR POLLUTANTS—
Continued**

[(NESHAPS) for Region VIII]

Subpart	State					
	CO	MT	ND	SD	UT	WY
O Inorganic Arsenic Emissions from Primary Copper Smelters.....		(*)			(*)	
P Inorganic Arsenic Emissions from Arsenic Trioxide and Metallic Arsenic Production Facilities.....		(*)			(*)	
V Equipment Leaks (Fugitive Emission Sources).....		(*)	(*)		(*)	

(*) Indicates delegation.

(1) Delegation for asbestos demolition, renovation and spraying operations, and waste disposal for demolition, renovation and spraying operations only.

[40 FR 18170, Apr. 25, 1975]

EDITORIAL NOTE: For FEDERAL REGISTER citations to § 61.04 see the List of CFR Sections Affected appearing in the Finding Aids section of this volume.

§ 61.05 Prohibited activities.

(a) After the effective date of any standard, no owner or operator shall construct or modify any stationary source subject to that standard without first obtaining written approval from the Administrator in accordance with this subpart, except under an exemption granted by the President under section 112(c)(2) of the Act. Sources, the construction or modification of which commenced after the publication date of the standards proposed to be applicable to the sources, are subject to this prohibition.

(b) After the effective date of any standard, no owner or operator shall operate a new stationary source subject to that standard in violation of the standard, except under an exemption granted by the President under section 112(c)(2) of the Act.

(c) Ninety days after the effective date of any standard, no owner or operator shall operate any existing source subject to that standard in violation of the standard, except under a waiver granted by the Administrator under this part or under an exemption granted by the President under section 112(c)(2) of the Act.

(d) No owner or operator subject to the provisions of this part shall fail to report, revise reports, or report source test results as required under this part.

[38 FR 8826, Apr. 6, 1973, as amended at 50 FR 46291, Nov. 7, 1985]

§ 61.06 Determination of construction or modification.

An owner or operator may submit to the Administrator a written application for a determination of whether actions intended to be taken by the owner or operator constitute construction or modification, or commencement thereof, of a source subject to a standard. The Administrator will notify the owner or operator of his determination within 30 days after receiving sufficient information to evaluate the application.

[50 FR 46291, Nov. 7, 1985]

§ 61.07 Application for approval of construction or modification.

(a) The owner or operator shall submit to the Administrator an application for approval of the construction of any new source or modification of any existing source. The application shall be submitted before the construction or modification is planned to commence, or within 30 days after the effective date if the construction or modification had commenced before the effective date and initial startup has not occurred. A separate application shall be submitted for each stationary source.

(b) Each application for approval of construction shall include—

- (1) The name and address of the applicant;
- (2) The location or proposed location of the source; and

(3) Technical information describing the proposed nature, size, design, operating design capacity, and method of operation of the source, including a description of any equipment to be used for control of emissions. Such technical information shall include calculations of emission estimates in sufficient detail to permit assessment of the validity of the calculations.

(c) Each application for approval of modification shall include, in addition to the information required in paragraph (b) of this section—

(1) The precise nature of the proposed changes;

(2) The productive capacity of the source before and after the changes are completed; and

(3) Calculations of estimates of emissions before and after the changes are completed, in sufficient detail to permit assessment of the validity of the calculations.

[50 FR 46291, Nov. 7, 1985]

§ 61.08 Approval of construction or modification.

(a) The Administrator will notify the owner or operator of approval or intention to deny approval of construction or modification within 60 days after receipt of sufficient information to evaluate an application under § 61.07.

(b) If the Administrator determines that a stationary source for which an application under § 61.07 was submitted will not cause emissions in violation of a standard if properly operated, the Administrator will approve the construction or modification.

(c) Before denying any application for approval of construction or modification, the Administrator will notify the applicant of the Administrator's intention to issue the denial together with—

(1) Notice of the information and findings on which the intended denial is based; and

(2) Notice of opportunity for the applicant to present, within such time limit as the Administrator shall specify, additional information or arguments to the Administrator before final action on the application.

(d) A final determination to deny any application for approval will be in

writing and will specify the grounds on which the denial is based. The final determination will be made within 60 days of presentation of additional information or arguments, or 60 days after the final date specified for presentation if no presentation is made.

(e) Neither the submission of an application for approval nor the Administrator's approval of construction or modification shall—

(1) Relieve an owner or operator of legal responsibility for compliance with any applicable provisions of this part or of any other applicable Federal, State, or local requirement; or

(2) Prevent the Administrator from implementing or enforcing this part or taking any other action under the Act.

[50 FR 46291, Nov. 7, 1985]

§ 61.09 Notification of startup.

(a) The owner or operator of each stationary source which has an initial startup after the effective date of a standard shall furnish the Administrator with written notification as follows:

(1) A notification of the anticipated date of initial startup of the source not more than 60 days nor less than 30 days before that date.

(2) A notification of the actual date of initial startup of the source within 15 days after that date.

(b) If any State or local agency requires a notice which contains all the information required in the notification in paragraph (a) of this section, sending the Administrator a copy of that notification will satisfy paragraph (a) of this section.

[50 FR 46291, Nov. 7, 1985]

§ 61.10 Source reporting and waiver request.

(a) The owner or operator of each existing source or each new source which had an initial startup before the effective date shall provide the following information in writing to the Administrator within 90 days after the effective date:

(1) Name and address of the owner or operator.

(2) The location of the source.

(3) The type of hazardous pollutants emitted by the stationary source.

(4) A brief description of the nature, size, design, and method of operation of the stationary source including the operating design capacity of the source. Identify each point of emission for each hazardous pollutant.

(5) The average weight per month of the hazardous materials being processed by the source, over the last 12 months preceding the date of the report.

(6) A description of the existing control equipment for each emission point including—

(i) Each control device for each hazardous pollutant; and

(ii) Estimated control efficiency (percent) for each control device.

(7) A statement by the owner or operator of the source as to whether the source can comply with the standards within 90 days after the effective date.

(b) The owner or operator of an existing source unable to comply with an applicable standard may request a waiver of compliance with that standard for a period not exceeding 2 years after the effective date. Any request shall be in writing and shall include the following information:

(1) A description of the controls to be installed to comply with the standard.

(2) A compliance schedule, including the date each step toward compliance will be reached. The list shall include as a minimum the following dates:

(i) Date by which contracts for emission control systems or process changes for emission control will be awarded, or date by which orders will be issued for the purchase of component parts to accomplish emission control or process changes;

(ii) Date of initiation of onsite construction or installation of emission control equipment or process change;

(iii) Date by which onsite construction or installation of emission control equipment or process change is to be completed; and

(iv) Date by which final compliance is to be achieved.

(3) A description of interim emission control steps which will be taken during the waiver period.

(c) Any change in the information provided under paragraph (a) of this section or § 61.07(b) shall be provided to the Administrator within 30 days after the change. However, if any change will result from modification of the source, §§ 61.07(c) and 61.08 apply.

(d) A possible format for reporting under this section is included as Appendix A of this part. Advice on reporting the status of compliance may be obtained from the Administrator.

[38 FR 8826, Apr. 6, 1973, as amended at 50 FR 46292, Nov. 7, 1985]

§ 61.11 Waiver of compliance.

(a) Based on the information provided in any request under § 61.10, or other information, the Administrator may grant a waiver of compliance with a standard for a period not exceeding 2 years after the effective date of the standard.

(b) The waiver will be in writing and will—

(1) Identify the stationary source covered;

(2) Specify the termination date of the waiver;

(3) Specify dates by which steps toward compliance are to be taken; and

(4) Specify any additional conditions which the Administrator determines necessary to assure installation of the necessary controls within the waiver period and to assure protection of the health of persons during the waiver period.

(c) The Administrator may terminate the waiver at an earlier date than specified if any specification under paragraphs (b)(3) and (b)(4) of this section are not met.

(d) Before denying any request for a waiver, the Administrator will notify the owner or operator making the request of the Administrator's intention to issue the denial, together with—

(1) Notice of the information and findings on which the intended denial is based; and

(2) Notice of opportunity for the owner or operator to present, within the time limit the Administrator specifies, additional information or argu-

ments to the Administrator before final action on the request.

(e) A final determination to deny any request for a waiver will be in writing and will set forth the specific grounds on which the denial is based. The final determination will be made within 60 days after presentation of additional information or argument; or within 60 days after the final date specified for the presentation if no presentation is made.

(f) The granting of a waiver under this section shall not abrogate the Administrator's authority under section 114 of the Act.

[50 FR 46292, Nov. 7, 1985]

§ 61.12 Compliance with standards and maintenance requirements.

(a) Compliance with numerical emission limits shall be determined by emission tests established in § 61.13 unless otherwise specified in an individual subpart.

(b) Compliance with design, equipment, work practice or operational standards shall be determined as specified in an individual subpart.

(c) The owner or operator of each stationary source shall maintain and operate the source, including associated equipment for air pollution control, in a manner consistent with good air pollution control practice for minimizing emissions. Determination of whether acceptable operating and maintenance procedures are being used will be based on information available to the Administrator which may include, but is not limited to, monitoring results, review of operating and maintenance procedures, and inspection of the source.

(d)(1) If, in the Administrator's judgment, an alternative means of emission limitation will achieve a reduction in emissions of a pollutant from a source at least equivalent to the reduction in emissions of that pollutant from that source achieved under any design, equipment, work practice or operational standard, the Administrator will publish in the FEDERAL REGISTER a notice permitting the use of the alternative means for purposes of compliance with the standard. The notice will restrict the permission to the source(s) or category(ies) of

sources on which the alternative means will achieve equivalent emission reductions. The notice may condition permission on requirements related to the operation and maintenance of the alternative means.

(2) Any notice under paragraph (d)(1) shall be published only after notice and an opportunity for a hearing.

(3) Any person seeking permission under this subsection shall, unless otherwise specified in the applicable subpart, submit a proposed test plan or the results of testing and monitoring, a description of the procedures followed in testing or monitoring, and a description of pertinent conditions during testing or monitoring.

[50 FR 46292, Nov. 7, 1985]

§ 61.13 Emission tests and waiver of emission tests.

(a) If required to do emission testing by an applicable subpart and unless a waiver of emission testing is obtained under this section, the owner or operator shall test emissions from the source—

(1) Within 90 days after the effective date, for an existing source or a new source which has an initial startup date before the effective date; or

(2) Within 90 days after initial startup, for a new source which has an initial startup date after the effective date.

(b) The Administrator may require an owner or operator to test emissions from the source at any other time when the action is authorized by section 114 of the Act.

(c) The owner or operator shall notify the Administrator of the emission test at least 30 days before the emission test to allow the Administrator the opportunity to have an observer present during the test.

(d) If required to do emission testing, the owner or operator of each new source and, at the request of the Administrator, the owner or operator of each existing source shall provide emission testing facilities as follows:

(1) Sampling ports adequate for test methods applicable to each source.

(2) Safe sampling platform(s).

(3) Safe access to sampling platform(s).

(4) Utilities for sampling and testing equipment.

(5) Any other facilities that the Administrator needs to safely and properly test a source.

(e) Each emission test shall be conducted under such conditions as the Administrator shall specify based on design and operational characteristics of the source.

(f) Unless otherwise specified in an applicable subpart, samples shall be analyzed and emissions determined within 30 days after each emission test has been completed. The owner or operator shall report the determinations of the emission test to the Administrator by a registered letter sent before the close of business on the 31st day following the completion of the emission test.

(g) The owner or operator shall retain at the source and make available, upon request, for inspection by the Administrator, for a minimum of 2 years, records of emission test results and other data needed to determine emissions.

(h)(1) Emission tests shall be conducted as set forth in this section, the applicable subpart and Appendix B unless the Administrator—

(i) Specifies or approves the use of a reference method with minor changes in methodology; or

(ii) Approves the use of an alternative method; or

(iii) Waives the requirement for emission testing because the owner or operator of a source has demonstrated by other means to the Administrator's satisfaction that the source is in compliance with the standard.

(2) If the Administrator finds reasonable grounds to dispute the results obtained by an alternative method, he may require the use of a reference method. If the results of the reference and alternative methods do not agree, the results obtained by the reference method prevail.

(3) The owner or operator may request approval for the use of an alternative method at any time, except—

(i) For an existing source or a new source that had an initial startup before the effective date, any request

for use of an alternative method during the initial emission test shall be submitted to the Administrator within 30 days after the effective date, or with the request for a waiver of compliance if one is submitted under § 60.10(b); or

(ii) For a new source that has an initial startup after the effective date, any request for use of an alternative method during the initial emission test shall be submitted to the Administrator no later than with the notification of anticipated startup required under § 60.09.

(1)(1) Emission tests may be waived upon written application to the Administrator if, in the Administrator's judgment, the source is meeting the standard, or the source is being operated under a waiver or compliance, or the owner or operator has requested a waiver of compliance and the Administrator is still considering that request.

(2) If application for waiver of the emission test is made, the application shall accompany the information required by § 61.10 or the notification of startup required by § 61.09, whichever is applicable. A possible format is contained in Appendix A to this part.

(3) Approval of any waiver granted under this section shall not abrogate the Administrator's authority under the Act or in any way prohibit the Administrator from later cancelling the waiver. The cancellation will be made only after notice is given to the owner or operator of the source.

[50 FR 46292, Nov. 7, 1985]

§ 61.14 Monitoring requirements.

(a) Unless otherwise specified, this section applies to each monitoring system required under each subpart which requires monitoring.

(b) Each owner or operator shall maintain and operate each monitoring system as specified in the applicable subpart and in a manner consistent with good air pollution control practice for minimizing emissions. Any unavoidable breakdown or malfunction of the monitoring system should be repaired or adjusted as soon as practicable after its occurrence. The Administrator's determination of whether acceptable operating and maintenance

procedures are being used will be based on information which may include, but not be limited to, review of operating and maintenance procedures, manufacturer recommendations and specifications, and inspection of the monitoring system.

(c) When required by the applicable subpart, and at any other time the Administrator may require, the owner or operator of a source being monitored shall conduct a performance evaluation of the monitoring system and furnish the Administrator with a copy of a written report of the results within 60 days of the evaluation. Such a performance evaluation shall be conducted according to the applicable specifications and procedures described in the applicable subpart. The owner or operator of the source shall furnish the Administrator with written notification of the date of the performance evaluation at least 30 days before the evaluation is to begin.

(d) When the effluents from a single source, or from two or more sources subject to the same emission standards, are combined before being released to the atmosphere, the owner or operator shall install a monitoring system on each effluent or on the combined effluent. If two or more sources are not subject to the same emission standards, the owner or operator shall install a separate monitoring system on each effluent, unless otherwise specified. If the applicable standard is a mass emission standard and the effluent from one source is released to the atmosphere through more than one point, the owner or operator shall install a monitoring system at each emission point unless the installation of fewer systems is approved by the Administrator.

(e) The owner or operator of each monitoring system shall reduce the monitoring data as specified in each applicable subpart. Monitoring data recorded during periods of unavoidable monitoring system breakdowns, repairs, calibration checks, and zero and span adjustments shall not be included in any data average.

(f) The owner or operator shall maintain records of monitoring data, monitoring system calibration checks, and the occurrence and duration of

any period during which the monitoring system is malfunctioning or inoperative. These records shall be maintained at the source for a minimum of 2 years and made available, upon request, for inspection by the Administrator.

(g)(1) Monitoring shall be conducted as set forth in this section and the applicable subpart unless the Administrator—

(i) Specifies or approves the use of the specified monitoring requirements and procedures with minor changes in methodology; or

(ii) Approves the use of alternatives to any monitoring requirements or procedures.

(2) If the Administrator finds reasonable grounds to dispute the results obtained by an alternative monitoring method, the Administrator may require the monitoring requirements and procedures specified in this part.

[50 FR 46293, Nov. 7, 1985]

§ 61.15 Modification.

(a) Except as provided under paragraph (d) of this section, any physical or operational change to a stationary source which results in an increase in the rate of emission to the atmosphere of a hazardous pollutant to which a standard applies shall be considered a modification.

(b) Upon modification, an existing source shall become a new source for each hazardous pollutant for which the rate of emission to the atmosphere increases and to which a standard applies.

(c) Emission rate shall be expressed as kg/hr of any hazardous pollutant discharged into the atmosphere for which a standard is applicable. The Administrator shall use the following to determine the emission rate:

(1) Emission factors as specified in the background information document (BID) for the applicable standard, or in the latest issue of "Compilation of Air Pollutant Emission Factors," EPA Publication No. AP-42, or other emission factors determined by the Administrator to be superior to AP-42 emission factors, in cases where use of emission factors demonstrates that the emission rate will clearly increase

or clearly not increase as a result of the physical or operational change.

(2) Material balances, monitoring data, or manual emission tests in cases where use of emission factors, as referenced in paragraph (c)(1) of this section, does not demonstrate to the Administrator's satisfaction that the emission rate will clearly increase or clearly not increase as a result of the physical or operational change, or where an interested person demonstrates to the Administrator's satisfaction that there are reasonable grounds to dispute the result obtained by the Administrator using emission factors. When the emission rate is based on results from manual emission tests or monitoring data, the procedures specified in Appendix C of 40 CFR part 60 shall be used to determine whether an increase in emission rate has occurred. Tests shall be conducted under such conditions as the Administrator shall specify to the owner or operator. At least three test runs must be conducted before and at least three after the physical or operational change. If the Administrator approves, the results of the emission tests required in § 61.13(a) may be used for the test runs to be conducted before the physical or operational change. All operating parameters which may affect emissions must be held constant to the maximum degree feasible for all test runs.

(d) The following shall not, by themselves, be considered modifications under this part:

(1) Maintenance, repair, and replacement which the Administrator determines to be routine for a source category.

(2) An increase in production rate of a stationary source, if that increase can be accomplished without a capital expenditure on the stationary source.

(3) An increase in the hours of operation.

(4) Any conversion to coal that meets the requirements specified in section 111(a)(8) of the Act.

(5) The relocation or change in ownership of a stationary source. However, such activities must be reported in accordance with § 61.10(c).

[50 FR 46294, Nov. 7, 1985]

§ 61.16 Availability of information.

The availability to the public of information provided to, or otherwise obtained by, the Administrator under this part shall be governed by Part 2 of this chapter.

[38 FR 8826, Apr. 6, 1973. Redesignated at 50 FR 46294, Nov. 7, 1985]

§ 61.17 State authority.

(a) This part shall not be construed to preclude any State or political subdivision thereof from—

(1) Adopting and enforcing any emission limiting regulation applicable to a stationary source, provided that such emission limiting regulation is not less stringent than the standards prescribed under this part; or

(2) Requiring the owner or operator of a stationary source to obtain permits, licenses, or approvals prior to initiating construction, modification, or operation of the source.

[50 FR 46294, Nov. 7, 1985]

§ 61.18 Incorporations by reference.

The materials listed below are incorporated by reference in the corresponding sections noted. These incorporations by reference were approved by the Director of the Federal Register on the date listed. These materials are incorporated as they exist on the date of the approval, and a notice of any change in these materials will be published in the **FEDERAL REGISTER**. The materials are available for purchase at the corresponding address noted below, and all are available for inspection at the Office of the Federal Register, Room 8401, 1100 L Street, NW., Washington, DC and the Library (MD-35), U.S. EPA, Research Triangle Park, North Carolina.

(a) The following material is available for purchase from at least one of the following addresses: American Society for Testing and Materials (ASTM), 1916 Race Street, Philadelphia, Pennsylvania 19103; or University Microfilms International, 300 North Zeeb Road, Ann Arbor, Michigan 48106.

(1) ASTM D737-75, Standard Test Method for Air Permeability of Textile Fabrics, in

corporation by reference (IBR) approved January 27, 1983 for § 61.23(a).

(2) ASTM D 1193-77, Standard Specification for Reagent Water, IBR approved for Method 101, par. 6.1.1; Method 101A, par. 6.1.1; Method 104, par. 3.1.2.

(3) ASTM D 2986-71 (Reapproved 1978), Standard Method for Evaluation of Air, Assay Media by the Monodisperse DOP (Diocetyl Phthalate) Smoke Test, IBR approved for Method 103, par. 2.1.3; Method 104, par. 3.1.1.

(4) ASTM D 2267-68 (reapproved 1978) Aromatics in Light Naphthas and Aviation Gasoline by Gas Chromatography, IBR approved June 6, 1984, for § 61.245(d)(1) and IBR approved September 30, 1986 for § 61.67(h)(1).

(5) ASTM D 2382-76, Heat of Combustion of Hydrocarbon Fuels by Bomb Calorimeter (High-Precision Method), IBR approved June 6, 1984, for § 61.245(e)(3).

(6) ASTM D 2504-67 (Reapproved 1977), Noncondensable Gases in C₃ and Lighter Hydrocarbon Products by Gas Chromatography, IBR approved June 6, 1984, for § 61.245(e)(3).

(7) ASTM D 836-84, Standard Specification for Industrial Grade Benzene, IBR approved September 14, 1989 for § 61.270(a).

EDITORIAL NOTE: The following paragraph (a)(7) was added to § 61.18 at 55 FR 22027, May 31, 1990 and will be correctly designated by an EPA document published in the FEDERAL REGISTER at a later date.

(7) ASTM E 50 (reapproved 1986), Standard Practices for Apparatus Reagents, and Safety Precautions for Chemical Analysis of Metals, IBR approved for Method 108C, par. 2.1.4.

(8) ASTM D 835-85, Standard Specification for Refined Benzene-485, IBR approved September 14, 1989 for § 61.270(a).

(9) ASTM D 2359-85a, Standard Specification for Refined Benzene-535, IBR approved September 14, 1989 for § 61.270(a).

(10) ASTM D 4734-87, Standard Specification for Refined Benzene-545, IBR approved September 14, 1989 for § 61.270(a).

(b) The following material is available from the U.S. EPA Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268.

(1) Method 601, Test Method for Purgeable Halocarbons, July 1982, IBR approved September 30, 1986 for § 61.67(g)(2).

(c) The following material is available for purchase from the American National Standards Institute, Inc., 1430 Broadway, New York, NY 10018.

(1) ANSI N13.1-1969, "Guide to Sampling Airborne Radioactive Materials in Nuclear Facilities." IBR approved for § 61.93(b)(2)(ii);

61.107(b)(2)(ii); and Method 114, par. 2.1 of Appendix B to part 61.

(d) The following material is available from the Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402-9325, telephone (202) 783-3238.

(1) Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, EPA Publication SW-846, Third Edition, November 1986, as amended by Revision I, December 1987, Order Number 955-001-00000-1:

(i) Method 8020, Aromatic Volatile Organics, IBR approved March 7, 1990, for § 61.355(c)(2)(iv)(A).

(ii) Method 8021, Volatile Organic Compounds in Water by Purge and Trap Capillary Column Gas Chromatography with Photoionization and Electrolytic Conductivity Detectors in Series, IBR approved March 7, 1990, for § 61.355(c)(2)(iv)(B).

(iii) Method 8240, Gas Chromatography/Mass Spectrometry for Volatile Organics, IBR approved March 7, 1990, for § 61.355(c)(2)(iv)(C).

(iv) Method 8260, Gas Chromatography/Mass Spectrometry for Volatile Organics: Capillary Column Technique, IBR approved March 7, 1990, for § 61.355(c)(2)(iv)(D).

[48 FR 3740, Jan. 27, 1983, as amended at 48 FR 55266, Dec. 9, 1983; 49 FR 23520, June 6, 1984; 51 FR 34914, Sept. 30, 1986; 54 FR 38073, Sept. 14, 1989; 54 FR 51704, Dec. 15, 1989; 55 FR 8341, Mar. 7, 1990; 55 FR 18331, May 2, 1990; 55 FR 22027, May 31, 1990]

§ 61.19 Circumvention.

No owner or operator shall build, erect, install, or use any article machine, equipment, process, or method, the use of which conceals an emission which would otherwise constitute a violation of an applicable standard. Such concealment includes, but is not limited to, the use of gaseous dilutants to achieve compliance with a visible emissions standard, and the piecemeal carrying out of an operation to avoid coverage by a standard that applies only to operations larger than a specified size.

[40 FR 48299, Oct. 14, 1975. Redesignated at 50 FR 46294, Nov. 7, 1985]

Subpart B—National Emission Standards for Radon Emissions From Underground Uranium Mines

SOURCE: 54 FR 51694, Dec. 15, 1989, unless otherwise noted.

§ 61.20

§ 61.20 Designation of facilities.

The provisions of this subpart are applicable to the owner or operator of an active underground uranium mine which:

(a) Has mined, will mine or is designed to mine over 100,000 tons of ore during the life of the mine; or

(b) Has had or will have an annual ore production rate greater than 10,000 tons, unless it can be demonstrated to EPA that the mine will not exceed total ore production of 100,000 tons during the life of the mine.

§ 61.21 Definitions.

As used in this subpart, all terms not defined here have the meaning given them in the Clean Air Act or subpart A of part 61. The following terms shall have the following specific meanings:

(a) *Active mine* means an underground uranium mine which is being ventilated to allow workers to enter the mine for any purpose.

(b) *Effective dose equivalent* means the sum of the products of absorbed dose and appropriate factors to account for differences in biological effectiveness due to the quality of radiation and its distribution in the body of reference man. The unit of the effective dose equivalent is the rem. The method for calculating effective dose equivalent and the definition of reference man are outlined in the International Commission on Radiological Protection's Publication No. 26.

(c) *Underground uranium mine* means a man-made underground excavation made for the purpose of removing material containing uranium for the principal purpose of recovering uranium.

§ 61.22 Standard.

Emissions of radon-222 to the ambient air from an underground uranium mine shall not exceed those amounts that would cause any member of the public to receive in any year an effective dose equivalent of 10 mrem/y.

§ 61.23 Determining compliance.

(a) Compliance with the emission standard in this subpart shall be determined and the effective dose equivalent calculated by the EPA computer code COMPLY-R. An underground

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uranium mine owner or operator shall calculate the source terms to be used for input into COMPLY-R by conducting testing in accordance with the procedures described in Appendix B, Method 115, or

(b) Owners or operators may demonstrate compliance with the emission standard in this subpart through the use of computer models that are equivalent to COMPLY-R provided that the model has received prior approval from EPA headquarters. EPA may approve a model in whole or in part and may limit its use to specific circumstances.

§ 61.24 Annual Reporting Requirements.

(a) The mine owner or operator shall annually calculate and report the results of the compliance calculations in section 61.23 and the input parameters used in making the calculation. Such report shall cover the emissions of a calendar year and shall be sent to EPA by March 31 of the following year. Each report shall also include the following information:

(1) The name and location of the mine.

(2) The name of the person responsible for the operation of the facility and the name of the person preparing the report (if different).

(3) The results of the emissions testing conducted and the dose calculated using the procedures in § 61.23.

(4) A list of the stacks or vents or other points where radioactive materials are released to the atmosphere, including their location, diameter, flow rate, effluent temperature and release height.

(5) A description of the effluent controls that are used on each stack, vent, or other release point and the effluent controls used inside the mine, and an estimate of the efficiency of each control method or device.

(6) Distances from the points of release to the nearest residence, school, business or office and the nearest farms producing vegetables, milk, and meat.

(7) The values used for all other user-supplied input parameters for the computer models (e.g., meteorological data) and the source of these data.

(8) Each report shall be signed and dated by a corporate officer in charge of the facility and contain the following declaration immediately above the signature line: "I certify under penalty of law that I have personally examined and am familiar with the information submitted herein and based on my inquiry of those individuals immediately responsible for obtaining the information, I believe that the submitted information is true, accurate and complete. I am aware that there are significant penalties for submitting false information including the possibility of fine and imprisonment. See, 18 U.S.C. 1001."

(b) If the facility is not in compliance with the emission standard of § 61.22 in the calendar year covered by the report, the facility must then commence reporting to the Administrator on a monthly basis the information listed in paragraph (a) of this section for the preceding month. These reports will start the month immediately following the submittal of the annual report for the year in noncompliance and will be due 30 days following the end of each month. This increased level of reporting will continue until the Administrator has determined that the monthly reports are no longer necessary. In addition to all the information required in paragraph (a) of this section, monthly reports shall also include the following information:

(1) All controls or other changes in operation of the facility that will be or are being installed to bring the facility into compliance.

(2) If the facility is under a judicial or administrative enforcement decree the report will describe the facilities performance under the terms of the decree.

(c) The first report will cover the emissions of calendar year 1990.

(Approved by the Office of Management and Budget under control number 2060-0191)

§ 61.25 Recordkeeping requirements.

The owner or operator of a mine must maintain records documenting the source of input parameters including the results of all measurements upon which they are based, the calculations and/or analytical methods

used to derive values for input parameters, and the procedure used to determine compliance. In addition, the documentation should be sufficient to allow an independent auditor to verify the accuracy of the determination made concerning the facility's compliance with the standard. These records must be kept at the mine or by the owner or operator for at least five years and upon request be made available for inspection by the Administrator, or his authorized representative.

§ 61.26 Exemption from the reporting and testing requirements of 40 CFR 61.10.

All facilities designated under this subpart are exempt from the reporting requirements of 40 CFR 61.10.

Subpart C—National Emission Standard for Beryllium

§ 61.30 Applicability.

The provisions of this subpart are applicable to the following stationary sources:

(a) Extraction plans, ceramic plants, foundries, incinerators, and propellant plants which process beryllium ore, beryllium, beryllium oxide, beryllium alloys, or beryllium-containing waste.

(b) Machine shops which process beryllium, beryllium oxides, or any alloy when such alloy contains more than 5 percent beryllium by weight.

§ 61.31 Definitions.

Terms used in this subpart are defined in the act, in subpart A of this part, or in this section as follows:

(a) *Beryllium* means the element beryllium. Where weights or concentrations are specified, such weights or concentrations apply to beryllium only, excluding the weight or concentration of any associated elements.

(b) *Extraction plant* means a facility chemically processing beryllium ore to beryllium metal, alloy, or oxide, or performing any of the intermediate steps in these processes.

(c) *Beryllium ore* means any naturally occurring material mined or gathered for its beryllium content.

(d) *Machine shop* means a facility performing cutting, grinding, turning, honing, milling, deburring, lapping,

electrochemical machining, etching, or other similar operations.

(e) *Ceramic plant* means a manufacturing plant producing ceramic items.

(f) *Foundry* means a facility engaged in the melting or casting of beryllium metal or alloy.

(g) *Beryllium-containing waste* means material contaminated with beryllium and/or beryllium compounds used or generated during any process or operation performed by a source subject to this subpart.

(h) *Incinerator* means any furnace used in the process of burning waste for the primary purpose of reducing the volume of the waste by removing combustible matter.

(i) *Propellant* means a fuel and oxidizer physically or chemically combined which undergoes combustion to provide rocket propulsion.

(j) *Beryllium alloy* means any metal to which beryllium has been added in order to increase its beryllium content and which contains more than 0.1 percent beryllium by weight.

(k) *Propellant plant* means any facility engaged in the mixing, casting, or machining of propellant.

§ 61.32 Emission standard.

(a) Emissions to the atmosphere from stationary sources subject to the provisions of this subpart shall not exceed 10 grams of beryllium over a 24-hour period, except as provided in paragraph (b) of this section.

(b) Rather than meet the requirement of paragraph (a) of this section, an owner or operator may request approval from the Administrator to meet an ambient concentration limit on beryllium in the vicinity of the stationary source of $0.01 \mu\text{g}/\text{m}^3$, averaged over a 30-day period.

(1) Approval of such requests may be granted by the Administrator provided that:

(i) At least 3 years of data is available which in the judgment of the Administrator demonstrates that the future ambient concentrations of beryllium in the vicinity of the stationary source will not exceed $0.01 \mu\text{g}/\text{m}^3$, averaged over a 30-day period. Such 3-year period shall be the 3 years ending 30 days before the effective date of this standard.

(ii) The owner or operator requests such approval in writing within 30 days after the effective date of this standard.

(iii) The owner or operator submits a report to the Administrator within 45 days after the effective date of this standard which report includes the following information:

(a) Description of sampling method including the method and frequency of calibration.

(b) Method of sample analysis.

(c) Averaging technique for determining 30-day average concentrations.

(d) Number, identity, and location (address, coordinates, or distance and heading from plant) of sampling sites.

(e) Ground elevations and height above ground of sampling inlets.

(f) Plant and sampling area plots showing emission points and sampling sites. Topographic features significantly affecting dispersion including plant building heights and locations shall be included.

(g) Information necessary for estimating dispersion including stack height, inside diameter, exit gas temperature, exit velocity or flow rate, and beryllium concentration.

(h) A description of data and procedures (methods or models) used to design the air sampling network (i.e., number and location of sampling sites).

(i) Air sampling data indicating beryllium concentrations in the vicinity of the stationary source for the 3-year period specified in paragraph (b)(1) of this section. This data shall be presented chronologically and include the beryllium concentration and location of each individual sample taken by the network and the corresponding 30-day average beryllium concentrations.

(2) Within 60 days after receiving such report, the Administrator will notify the owner or operator in writing whether approval is granted or denied. Prior to denying approval to comply with the provisions of paragraph (b) of this section, the Administrator will consult with representatives of the statutory source for which the demonstration report was submitted.

(c) The burning of beryllium and/or beryllium-containing waste, except

propellants, is prohibited except in incinerators, emissions from which must comply with the standard.

§ 61.33 Stack sampling.

(a) Unless a waiver of emission testing is obtained under § 61.13, each owner or operator required to comply with § 61.32(a) shall test emissions from the source according to Method 104 of Appendix B to this part. Method 103 of Appendix B to this part is approved by the Administrator as an alternative method for sources subject to § 61.32(a). The emission test shall be performed—

(1) Within 90 days of the effective date in the case of an existing source or a new source which has an initial startup date preceding the effective date; or

(2) Within 90 days of startup in the case of a new source which did not have an initial startup date preceding the effective date.

(b) The Administrator shall be notified at least 30 days prior to an emission test so that he may at his option observe the test.

(c) Samples shall be taken over such a period or periods as are necessary to accurately determine the maximum emissions which will occur in any 24-hour period. Where emissions depend upon the relative frequency of operation of different types of processes, operating hours, operating capacities, or other factors, the calculation of maximum 24-hour-period emissions will be based on that combination of factors which is likely to occur during the subject period and which result in the maximum emissions. No changes in the operation shall be made, which would potentially increase emissions above that determined by the most recent source test, until a new emission level has been estimated by calculation and the results reported to the Administrator.

(d) All samples shall be analyzed and beryllium emissions shall be determined within 30 days after the source test. All determinations shall be reported to the Administrator by a registered letter dispatched before the close of the next business day following such determination.

(e) Records of emission test results and other data needed to determine total emissions shall be retained at the source and made available, for inspection by the Administrator, for a minimum of 2 years.

[38 FR 8826, Apr. 6, 1973, as amended at 50 FR 46294, Nov. 7, 1985]

§ 61.34 Air sampling.

(a) Stationary sources subject to § 61.32(b) shall locate air sampling sites in accordance with a plan approved by the Administrator. Such sites shall be located in such a manner as is calculated to detect maximum concentrations of beryllium in the ambient air.

(b) All monitoring sites shall be operated continuously except for a reasonable time allowance for instrument maintenance and calibration, for changing filters, or for replacement of equipment needing major repair.

(c) Filters shall be analyzed and concentrations calculated within 30 days after filters are collected. Records of concentrations at all sampling sites and other data needed to determine such concentrations shall be retained at the source and made available, for inspection by the Administrator, for a minimum of 2 years.

(d) Concentrations measured at all sampling sites shall be reported to the Administrator every 30 days by a registered letter.

(e) The Administrator may at any time require changes in, or expansion of, the sampling network.

Subpart D—National Emission Standard for Beryllium Rocket Motor Firing

§ 61.40 Applicability.

The provisions of this subpart are applicable to rocket motor test sites.

§ 61.41 Definitions.

Terms used in this subpart are defined in the Act, in subpart A of this part, or in this section as follows:

(a) "Rocket motor test site" means any building, structure, facility, or installation where the static test firing of a beryllium rocket motor and/or

the disposal of beryllium propellant is conducted.

(b) "Beryllium propellant" means any propellant incorporating beryllium.

§ 61.42 Emission standard.

(a) Emissions to the atmosphere from rocket-motor test sites shall not cause time-weighted atmospheric concentrations of beryllium to exceed 75 microgram minutes per cubic meter of air within the limits of 10 to 60 minutes, accumulated during any 2 consecutive weeks, in any area in which an effect adverse to public health could occur.

(b) If combustion products from the firing of beryllium propellant are collected in a closed tank, emissions from such tank shall not exceed 2 grams per hour and a maximum of 10 grams per day.

§ 61.43 Emission testing—rocket firing or propellant disposal.

(a) Ambient air concentrations shall be measured during and after firing of a rocket motor or propellant disposal and in such a manner that the effect of these emissions can be compared with the standard. Such sampling techniques shall be approved by the Administrator.

(b) All samples shall be analyzed and results shall be calculated within 30 days after samples are taken and before any subsequent rocket motor firing or propellant disposal at the given site. All results shall be reported to the Administrator by a registered letter dispatched before the close of the next business day following determination of such results.

(c) Records of air sampling test results and other data needed to determine integrated intermittent concentrations shall be retained at the source and made available, for inspection by the Administrator, for a minimum of 2 years.

(d) The Administrator shall be notified at least 30 days prior to an air sampling test, so that he may at his option observe the test.

§ 61.44 Stack sampling.

(a) Sources subject to § 61.42(b) shall be continuously sampled, during re-

lease of combustion products from the tank, according to Method 104 of Appendix B to this part. Method 103 of Appendix B to this part is approved by the Administrator as an alternative method for sources subject to § 61.42(b).

(b) All samples shall be analyzed, and beryllium emissions shall be determined within 30 days after samples are taken and before any subsequent rocket motor firing or propellant disposal at the given site. All determinations shall be reported to the Administrator by a registered letter dispatched before the close of the next business day following such determinations.

(c) Records of emission test results and other data needed to determine total emissions shall be retained at the source and made available, for inspection by the Administrator, for a minimum of 2 years.

(d) The Administrator shall be notified at least 30 days prior to an emission test, so that he may at his option observe the test.

[38 FR 8826, Apr. 6, 1973, as amended at 50 FR 46294, Nov. 7, 1985]

Subpart E—National Emission Standard for Mercury

§ 61.50 Applicability.

The provisions of this subpart are applicable to those stationary sources which process mercury ore to recover mercury, use mercury chlor-alkali cells to produce chlorine gas and alkali metal hydroxide, and incinerate or dry wastewater treatment plant sludge.

[40 FR 48302, Oct. 14, 1975]

§ 61.51 Definitions.

Terms used in this subpart are defined in the act, in subpart A of this part, or in this section as follows:

(a) "Mercury" means the element mercury, excluding any associated elements, and includes mercury in particulates, vapors, aerosols, and compounds.

(b) "Mercury ore" means a mineral mined specifically for its mercury content.

(c) "Mercury ore processing facility" means a facility processing mercury ore to obtain mercury.

(d) "Condenser stack gases" mean the gaseous effluent evolved from the stack of processes utilizing heat to extract mercury metal from mercury ore.

(e) "Mercury chlor-alkali cell" means a device which is basically composed of an electrolyzer section and a denuder (decomposer) section and utilizes mercury to produce chlorine gas, hydrogen gas, and alkali metal hydroxide.

(f) "Mercury chlor-alkali electrolyzer" means an electrolytic device which is part of a mercury chlor-alkali cell and utilizes a flowing mercury cathode to produce chlorine gas and alkali metal amalgam.

(g) "Denuder" means a horizontal or vertical container which is part of a mercury chlor-alkali cell and in which water and alkali metal amalgam are converted to alkali metal hydroxide, mercury, and hydrogen gas in a short-circuited, electrolytic reaction.

(h) "Hydrogen gas stream" means a hydrogen stream formed in the chlor-alkali cell denuder.

(i) "End box" means a container(s) located on one or both ends of a mercury chlor-alkali electrolyzer which serves as a connection between the electrolyzer and denuder for rich and stripped amalgam.

(j) "End box ventilation system" means a ventilation system which collects mercury emissions from the end-boxes, the mercury pump sumps, and their water collection systems.

(k) "Cell room" means a structure(s) housing one or more mercury electrolytic chlor-alkali cells.

(l) "Sludge" means sludge produced by a treatment plant that processes municipal or industrial waste waters.

(m) "Sludge dryer" means a device used to reduce the moisture content of sludge by heating to temperatures above 65°C (ca. 150°F) directly with combustion gases.

[38 FR 8826, Apr. 6, 1973, as amended at 40 FR 48302, Oct. 14, 1975]

§ 61.52 Emission standard.

(a) Emissions to the atmosphere from mercury ore processing facilities and mercury cell chlor-alkali plants

shall not exceed 2300 grams of mercury per 24-hour period.

(b) Emissions to the atmosphere from sludge incineration plants, sludge drying plants, or a combination of these that process wastewater treatment plant sludges shall not exceed 3200 grams of mercury per 24-hour period.

[40 FR 48302, Oct. 14, 1975]

§ 61.53 Stack sampling.

(a) *Mercury ore processing facility.*

(1) Unless a waiver of emission testing is obtained under § 61.13, each owner or operator processing mercury ore shall test emissions from the source according to Method 101 of Appendix B to this part. The emission test shall be performed—

(i) Within 90 days of the effective date in the case of an existing source or a new source which has an initial start-up date preceding the effective date; or

(ii) Within 90 days of startup in the case of a new source which did not have an initial startup date preceding the effective date.

(2) The Administrator shall be notified at least 30 days prior to an emission test, so that he may at his option observe the test.

(3) Samples shall be taken over such a period or periods as are necessary to accurately determine the maximum emissions which will occur in a 24-hour period. No changes in the operation shall be made, which would potentially increase emissions above that determined by the most recent source test, until the new emission level has been estimated by calculation and the results reported to the Administrator.

(4) All samples shall be analyzed and mercury emissions shall be determined within 30 days after the stack test. Each determination shall be reported to the Administrator by a registered letter dispatched within 15 calendar days following the date such determination is completed.

(5) Records of emission test results and other data needed to determine total emissions shall be retained at the source and made available, for inspection by the Administrator, for a minimum of 2 years.

(b) *Mercury chlor-alkali plant—hydrogen and end-box ventilation gas streams.* (1) Unless a waiver of emission testing is obtained under § 61.13, each owner or operator employing mercury chlor-alkali cell(s) shall test emissions from hydrogen streams according to Method 102 and from end-box ventilation gas streams according to Method 101 of Appendix B to this part. The emission test shall be performed—

(i) Within 90 days of the effective date in the case of an existing source or a new source which has an initial startup date preceding the effective date; or

(ii) Within 90 days of startup in the case of a new source which did not have an initial startup date preceding the effective date.

(2) The Administrator shall be notified at least 30 days prior to an emission test, so that he may at his option observe the test.

(3) Samples shall be taken over such a period or periods as are necessary to accurately determine the maximum emissions which will occur in a 24-hour period. No changes in the operation shall be made, which would potentially increase emissions above that determined by the most recent source test, until the new emission has been estimated by calculation and the results reported to the Administrator.

(4) All samples shall be analyzed and mercury emissions shall be determined within 30 days after the stack test. Each determination shall be reported to the Administrator by a registered letter dispatched within 15 calendar days following the date such determination is completed.

(5) Records of emission test results and other data needed to determine total emissions shall be retained at the source and made available, for inspection by the Administrator, for a minimum of 2 years.

(c) *Mercury chlor-alkali plants—cell room ventilation system.* (1) Stationary sources using mercury chlor-alkali cells may test cell room emissions in accordance with paragraph (c)(2) of this section or demonstrate compliance with paragraph (c)(4) of this section and assume ventilation emissions of 1,300 gms/day of mercury.

(2) Unless a waiver of emission testing is obtained under § 61.13, each owner or operator shall pass all cell room air in force gas streams through stacks suitable for testing and shall test emissions from the source according to Method 101 in Appendix B to this part. The emission test shall be performed—

(i) Within 90 days of the effective date in the case of an existing source or a new source which has an initial startup date preceding the effective date; or

(ii) Within 90 days of startup in the case of a new source which did not have an initial startup date preceding the effective date.

(3) The Administrator shall be notified at least 30 days prior to an emission test, so that he may at his option observe the test.

(4) An owner or operator may carry out approved design, maintenance, and housekeeping practices. A list of approved practices is provided in Appendix A of "Review of National Emission Standards for Mercury," EPA-450/3-84-014a, December 1984. Copies are available from EPA's Central Docket Section, Docket item number A-84-41, III-B-1.

(d) *Sludge incineration and drying plants.* (1) Unless a waiver of emission testing is obtained under § 61.13, each owner or operator of a source subject to the standard in § 61.52(b) shall test emissions from that source. Such tests shall be conducted in accordance with the procedures set forth either in paragraph (d) of this section or in § 61.54.

(2) Method 101A in Appendix B to this part shall be used to test emissions as follows:

(i) The test shall be performed within 90 days of the effective date of these regulations in the case of an existing source or a new source which has an initial startup date preceding the effective date.

(ii) The test shall be performed within 90 days of startup in the case of a new source which did not have an initial startup date preceding the effective date.

(3) The Administrator shall be notified at least 30 days prior to an emis-

sion test, so that he may at his option observe the test.

(4) Samples shall be taken over such a period or periods as are necessary to determine accurately the maximum emissions which will occur in a 24-hour period. No changes shall be made in the operation which would potentially increase emissions above the level determined by the most recent stack test, until the new emission level has been estimated by calculation and the results reported to the Administrator.

(5) All samples shall be analyzed and mercury emissions shall be determined within 30 days after the stack test. Each determination shall be reported to the Administrator by a registered letter dispatched within 15 calendar days following the date such determination is completed.

(6) Records of emission test results and other data needed to determine total emissions shall be retained at the source and shall be made available, for inspection by the Administrator, for a minimum of 2 years.

[38 FR 8826, Apr. 6, 1973, as amended at 40 FR 48302, Oct. 14, 1975; 47 FR 24704, June 8, 1982; 50 FR 46294, Nov. 7, 1985; 52 FR 8726, Mar. 19, 1987]

§ 61.54 Sludge sampling.

(a) As an alternative means for demonstrating compliance with § 61.52(b), an owner or operator may use Method 105 of Appendix B and the procedures specified in this section.

(1) A sludge test shall be conducted within 90 days of the effective date of these regulations in the case of an existing source or a new source which has an initial startup date preceding the effective date; or

(2) A sludge test shall be conducted within 90 days of startup in the case of a new source which did not have an initial startup date preceding the effective date.

(b) The Administrator shall be notified at least 30 days prior to a sludge sampling test, so that he may at his option observe the test.

(c) Sludge shall be sampled according to paragraph (c)(1) of this section, sludge charging rate for the plant shall be determined according to paragraph (c)(2) of this section, and the

sludge analysis shall be performed according to paragraph (c)(3) of this section.

(1) The sludge shall be sampled according to Method 105—Determination of Mercury in Wastewater Treatment Plant Sewage Sludges. A total of three composite samples shall be obtained within an operating period of 24 hours. When the 24-hour operating period is not continuous, the total sampling period shall not exceed 72 hours after the first grab sample is obtained. Samples shall not be exposed to any condition that may result in mercury contamination or loss.

(2) The maximum 24-hour period sludge incineration or drying rate shall be determined by use of a flow rate measurement device that can measure the mass rate of sludge charged to the incinerator or dryer with an accuracy of ± 5 percent over its operating range. Other methods of measuring sludge mass charging rates may be used if they have received prior approval by the Administrator.

(3) The sampling, handling, preparation, and analysis of sludge samples shall be accomplished according to Method 105 in Appendix B of this part.

(d) The mercury emissions shall be determined by use of the following equation.

$$E_{Hg} = \frac{MQ F_{sm(avg)}}{1000}$$

where:

E_{Hg} = Mercury emissions, g/day.

M = Mercury concentration of sludge on a dry solids basis, $\mu\text{g/g}$.

Q = Sludge charging rate, kg/day.

F_{sm} = Weight fraction of solids in the collected sludge after mixing.

1000 = Conversion factor, kg $\mu\text{g/g}^2$.

(e) No changes in the operation of a plant shall be made after a sludge test has been conducted which would potentially increase emissions above the level determined by the most recent sludge test, until the new emission level has been estimated by calculation and the results reported to the Administrator.

(f) All sludge samples shall be analyzed for mercury content within 30 days after the sludge sample is collected. Each determination shall be reported to the Administrator by a registered letter dispatched within 15 calendar days following the date such determination is completed.

(g) Records of sludge sampling, charging rate determination and other data needed to determine mercury content of wastewater treatment plant sludges shall be retained at the source and made available, for inspection by the Administrator, for a minimum of 2 years.

[40 FR 48303, Oct. 14, 1975, as amended at 49 FR 35770, Sept. 12, 1984; 52 FR 8727, Mar. 19, 1987; 53 FR 36972, Sept. 23, 1988]

§ 61.55 Monitoring of emissions and operations.

(a) Wastewater treatment plant sludge incineration and drying plants. All the sources for which mercury emissions exceed 1,600 g per 24-hour period, demonstrated either by stack sampling according to § 61.53 or sludge sampling according to § 61.54, shall monitor mercury emissions at intervals of at least once per year by use of Method 105 of Appendix B or the procedures specified in § 61.53 (d) (2) and (4). The results of monitoring shall be reported and retained according to § 61.53(d) (5) and (6) or § 61.54 (f) and (g).

(b) Mercury cell chlor-alkali plants—hydrogen and end-box ventilation gas streams.

(1) The owner or operator of each mercury cell chlor-alkali plant shall, within 1 year of the date of publication of these amendments or within 1 year of startup for a plant with initial startup after the date of publication, perform a mercury emission test that demonstrates compliance with the emission limits in § 61.52, on the hydrogen stream by Reference Method 102 and on the end-box stream by Reference Method 101 for the purpose of establishing limits for parameters to be monitored.

(2) During tests specified in paragraph (b)(1) of this section, the following control device parameters shall be monitored, except as provided in paragraph (c) of this section, and recorded

manually or automatically at least once every 15 minutes:

(i) The exit gas temperature from uncontrolled streams;

(ii) The outlet temperature of the gas stream from the final (i.e., the farthest downstream) cooling system when no control devices other than coolers and demisters are used;

(iii) The outlet temperature of the gas stream from the final cooling system when the cooling system is followed by a molecular sieve or carbon adsorber;

(iv) Outlet concentration of available chlorine, pH, liquid flow rate, and inlet gas temperature of chlorinated brine scrubbers and hypochlorite scrubbers;

(v) The liquid flow rate and exit gas temperature for water scrubbers;

(vi) The inlet gas temperature of carbon adsorption systems; and

(vii) The temperature during the heating phase of the regeneration cycle for carbon adsorbers or molecular sieves.

(3) The recorded parameters in paragraphs (b)(2)(i) through (b)(2)(vi) of this section shall be averaged over the test period (a minimum of 6 hours) to provide an average number. The highest temperature reading that is measured in paragraph (b)(2)(vii) of this section is to be identified as the reference temperature for use in paragraph (b)(6)(ii) of this section.

(4)(i) Immediately following completion of the emission tests specified in paragraph (b)(1) of this section, the owner or operator of a mercury cell chlor-alkali plant shall monitor and record manually or automatically at least once per hour the same parameters specified in paragraphs (b)(2)(i) through (b)(2)(vi) of this section.

(ii) Immediately following completion of the emission tests specified in paragraph (b)(1) of this section, the owner or operator shall monitor and record manually or automatically, during each heating phase of the regeneration cycle, the temperature specified in paragraph (b)(2)(vii) of this section.

(5) Monitoring devices used in accordance with paragraphs (b)(2) and (b)(4) of this section shall be certified by their manufacturer to be accurate

to within 10 percent, and shall be operated, maintained, and calibrated according to the manufacturer's instructions. Records of the certifications and calibrations shall be retained at the chlor-alkali plant and made available for inspection by the Administrator as follows: Certification, for as long as the device is used for this purpose; calibration for a minimum of 2 years.

(6)(i) When the hourly value of a parameter monitored in accordance with paragraph (b)(4)(i) of this section exceeds, or in the case of liquid flow rate and available chlorine falls below the value of that same parameter determined in paragraph (b)(2) of this section for 24 consecutive hours, the Administrator is to be notified within the next 10 days.

(ii) When the maximum hourly value of the temperature measured in accordance with paragraph (b)(4)(ii) of this section is below the reference temperature recorded according to paragraph (b)(3) of this section for three consecutive regeneration cycles, the Administrator is to be notified within the next 10 days.

(7) Semiannual reports shall be submitted to the Administrator indicating the time and date on which the hourly value of each parameter monitored according to paragraphs (b)(4)(i) and (b)(4)(ii) of this section fell outside the value of that same parameter determined under paragraph (b)(3) of this section; and corrective action taken, and the time and date of the corrective action. Parameter excursions will be considered unacceptable operation and maintenance of the emission control system. In addition, while compliance with the emission limits is determined primarily by conducting a performance test according to the procedures in § 61.53(b), reports of parameter excursions may be used as evidence in judging the duration of a violation that is determined by a performance test.

(8) Semiannual reports required in paragraph (b)(7) of this section shall be submitted to the Administrator on September 15 and March 15 of each year. The first semiannual report is to be submitted following the first full 6 month reporting period. The semiannual report due on September 15

(March 15) shall include all excursions monitored through August 31 (February 28) of the same calendar year.

(c) As an alternative to the monitoring, recordkeeping, and reporting requirements in paragraphs (b)(2) through (8) of this section, an owner or operator may develop and submit for the Administrator's review and approval a plant-specific monitoring plan. To be approved, such a plan must ensure not only compliance with the emission limits of § 61.52(a) but also proper operation and maintenance of emissions control systems. Any site-specific monitoring plan submitted must, at a minimum, include the following:

(1) Identification of the critical parameter or parameters for the hydrogen stream and for the end-box ventilation stream that are to be monitored and an explanation of why the critical parameter(s) selected is the best indicator of proper control system performance and of mercury emission rates.

(2) Identification of the maximum or minimum value of each parameter (e.g., degrees temperature, concentration of mercury) that is not to be exceeded. The level(s) is to be directly correlated to the results of a performance test, conducted no more than 180 days prior to submittal of the plan, when the facility was in compliance with the emission limits of § 61.52(a).

(3) Designation of the frequency for recording the parameter measurements, with justification if the frequency is less than hourly. A longer recording frequency must be justified on the basis of the amount of time that could elapse during periods of process or control system upsets before the emission limits would be exceeded, and consideration is to be given to the time that would be necessary to repair the failure.

(4) Designation of the immediate actions to be taken in the event of an excursion beyond the value of the parameter established in 2.

(5) Provisions for reporting, semiannually, parameter excursions and the corrective actions taken, and provisions for reporting within 10 days any significant excursion.

(6) Identification of the accuracy of the monitoring device(s) or of the readings obtained.

(7) Recordkeeping requirements for certifications and calibrations.

(d) Mercury cell chlor-alkali plants—cell room ventilation system.

(1) Stationary sources determining cell room emissions in accordance with § 61.53(c)(4) shall maintain daily records of all leaks or spills of mercury. The records shall indicate the amount, location, time, and date the leaks or spills occurred, identify the cause of the leak or spill, state the immediate steps taken to minimize mercury emissions and steps taken to prevent future occurrences, and provide the time and date on which corrective steps were taken.

(2) The results of monitoring shall be recorded, retained at the source, and made available for inspection by the Administrator for a minimum of 2 years.

(Approved by the Office of Management and Budget under control number 2060-0097)

[52 FR 8727, Mar. 19, 1987]

§ 61.56 Delegation of authority.

(a) In delegating implementation and enforcement authority to a State under section 112(d) of the Act, the authorities contained in paragraph (b) of this section shall be retained by the Administrator and not transferred to a State.

(b) Authorities which will not be delegated to States: Sections 61.53(c)(4) and 61.55(d). The authorities not delegated to States listed are in addition to the authorities in the General Provisions, subpart A of 40 CFR part 61, that will not be delegated to States (§§ 61.04(b), 61.12(d)(1), and 61.13(h)(1)(ii)).

[52 FR 8728, Mar. 19, 1987]

Subpart F—National Emission Standard for Vinyl Chloride

SOURCE: 41 FR 46564, Oct. 21, 1976, unless otherwise noted.

§ 61.60 Applicability.

(a) This subpart applies to plants which produce:

(1) Ethylene dichloride by reaction of oxygen and hydrogen chloride with ethylene.

(2) Vinyl chloride by any process, and/or

(3) One or more polymers containing any fraction of polymerized vinyl chloride.

(b) This subpart does not apply to equipment used in research and development if the reactor used to polymerize the vinyl chloride processed in the equipment has a capacity of no more than 0.19 m³ (50 gal).

(c) Sections of this subpart other than §§ 61.61; 61.64 (a)(1), (b), (c), and (d); 61.67; 61.68; 61.69; 61.70; and 61.71 do not apply to equipment used in research and development if the reactor used to polymerize the vinyl chloride processed in the equipment has a capacity of greater than 0.19 m³ (50 gal) and no more than 4.07 m³ (1075 gal).

[41 FR 46564, Oct. 21, 1976, as amended at 42 FR 29006, June 7, 1977; 53 FR 36972, Sept. 23, 1988]

§ 61.61 Definitions.

Terms used in this subpart are defined in the Act, in subpart A of this part, or in this section as follows:

(a) "Ethylene dichloride plant" includes any plant which produces ethylene dichloride by reaction of oxygen and hydrogen chloride with ethylene.

(b) "Vinyl chloride plant" includes any plant which produces vinyl chloride by any process.

(c) "Polyvinyl chloride plant" includes any plant where vinyl chloride alone or in combination with other materials is polymerized.

(d) "Slip gauge" means a gauge which has a probe that moves through the gas/liquid interface in a storage or transfer vessel and indicates the level of vinyl chloride in the vessel by the physical state of the material the gauge discharges.

(e) "Type of resin" means the broad classification of resin referring to the basic manufacturing process for producing that resin, including, but not limited to, the suspension, dispersion, latex, bulk, and solution processes.

(f) "Grade of resin" means the subdivision of resin classification which describes it as a unique resin, i.e., the

most exact description of a resin with no further subdivision.

(g) "Dispersion resin" means a resin manufactured in such a way as to form fluid dispersions when dispersed in a plasticizer or plasticizer/diluent mixtures.

(h) "Latex resin" means a resin which is produced by a polymerization process which initiates from free radical catalyst sites and is sold undried.

(i) "Bulk resin" means a resin which is produced by a polymerization process in which no water is used.

(j) "Inprocess wastewater" means any water which, during manufacturing or processing, comes into direct contact with vinyl chloride or polyvinyl chloride or results from the production or use of any raw material, intermediate product, finished product, by-product, or waste product containing vinyl chloride or polyvinyl chloride but which has not been discharged to a wastewater treatment process or discharged untreated as wastewater. Gas-holder seal water is not inprocess wastewater until it is removed from the gasholder.

(k) "Wastewater treatment process" includes any process which modifies characteristics such as BOD, COD, TSS, and pH, usually for the purpose of meeting effluent guidelines and standards; it does not include any process the purpose of which is to remove vinyl chloride from water to meet requirements of this subpart.

(l) "In vinyl chloride service" means that a piece of equipment either contains or contacts a liquid that is at least 10 percent vinyl chloride by weight or a gas that is at least 10 percent by volume vinyl chloride as determined according to the provisions of § 61.67(h). The provisions of § 61.67(h) also specify how to determine that a piece of equipment is not in vinyl chloride service. For the purposes of this subpart, this definition must be used in place of the definition of "in VHAP service" in subpart V of this part.

(m) "Standard operating procedure" means a formal written procedure officially adopted by the plant owner or operator and available on a routine basis to those persons responsible for carrying out the procedure.

(n) "Run" means the net period of time during which an emission sample is collected.

(o) "Ethylene dichloride purification" includes any part of the process of ethylene dichloride production which follows ethylene dichloride formation, excluding product storage following the final finishing column.

(p) "Vinyl chloride purification" includes any part of the process of vinyl chloride production which follows vinyl chloride formation.

(q) "Reactor" includes any vessel in which vinyl chloride is partially or totally polymerized into polyvinyl chloride.

(r) "Reactor opening loss" means the emissions of vinyl chloride occurring when a reactor is vented to the atmosphere for any purpose other than an emergency relief discharge as defined in § 61.65(a).

(s) "Stripper" includes any vessel in which residual vinyl chloride is removed from polyvinyl chloride resin, except bulk resin, in the slurry form by the use of heat and/or vacuum. In the case of bulk resin, stripper includes any vessel which is used to remove residual vinyl chloride from polyvinyl chloride resin immediately following the polymerization step in the plant process flow.

(t) "Standard temperature" means a temperature of 20° C (69° F).

(u) "Standard pressure" means a pressure of 760 mm of Hg (29.92 in. of Hg).

(v) "Relief valve" means each pressure relief device including pressure relief valves, rupture disks and other pressure relief systems used to protect process components from overpressure conditions. "Relief valve" does not include polymerization shortstop systems, refrigerated water systems or control valves or other devices used to control flow to an incinerator or other air pollution control device.

(w) "Leak" means any of several events that indicate interruption of confinement of vinyl chloride within process equipment. Leaks include events regulated under subpart V of this part such as:

(1) An instrument reading of 10,000 ppm or greater measure according to

Method 21 (see Appendix A of 40 CFR part 60);

(2) Indications of liquid dripping;

(3) A sensor detection of failure of a seal system, failure of a barrier fluid system, or both; and

(4) Detectable emissions as indicated by an instrument reading of greater than 500 ppm above background for equipment designated for no detectable emissions measured according to Test Method 21 (see Appendix A of 40 CFR part 60). Leaks also include events regulated under § 61.65(b)(8)(i) for detection of ambient concentrations in excess of background concentration. A relief valve discharge is not a leak.

(x) "Exhaust gas" means any offgas (the constituents of which may consist of any fluids, either as a liquid and/or gas) discharged directly or ultimately to the atmosphere that was initially contained in or was in direct contact with the equipment for which exhaust gas limits are prescribed in § 61.62 (a) and (b); § 61.63(a); § 61.64 (a)(1), (a)(2), (b), (c), and (d); § 61.65(b) (1)(ii), (b)(2), (b)(5), (b)(6)(ii) and (b)(9)(ii).

(y) "Relief valve discharge" means any nonleak discharge through a relief valve. "Relief valve discharge" does not include discharges ducted to a control system from which the concentration of vinyl chloride in the exhaust gases does not exceed 10 ppm (average for 3-hour period), or equivalent as provided in § 61.66.

(z) "3-hour period" means any three consecutive 1-hour periods (each hour commencing on the hour).

[41 FR 46564, Oct. 21, 1976, as amended at 42 FR 29006, June 7, 1977; 51 FR 34908, Sept. 30, 1986]

§ 61.62 Emission standard for ethylene dichloride plants.

(a) *Ethylene dichloride purification.* The concentration of vinyl chloride in each exhaust gas stream from any equipment used in ethylene dichloride purification is not to exceed 10 ppm (average for 3-hour period), except as provided in § 61.65(a). This requirement does not preclude combining of exhaust gas streams provided the combined steam is ducted through a control system from which the concentration of vinyl chloride in the exhaust

gases does not exceed 10 ppm, or equivalent as provided in § 61.66. This requirement does not apply to equipment that has been opened, is out of operation, and met the requirement in § 61.65(b)(6)(i) before being opened.

(b) *Orychlorination reactor.* Except as provided in § 61.65(a), emissions of vinyl chloride to the atmosphere from each oxychlorination reactor are not to exceed 0.2 g/kg (0.0002 lb/lb) (average for 3-hour period) of the 100 percent ethylene dichloride product from the oxychlorination process.

[51 FR 34909, Sept. 30, 1986]

§ 61.63 Emission standard for vinyl chloride plants.

An owner or operator of a vinyl chloride plant shall comply with the requirements of this section and § 61.65.

(a) Vinyl chloride formation and purification: The concentration of vinyl chloride in each exhaust gas stream from any equipment used in vinyl chloride formation and/or purification is not to exceed 10 ppm (average for 3-hour period), except as provided in § 61.65(a). This requirement does not preclude combining of exhaust gas streams provided the combined steam is ducted through a control system from which the concentration of vinyl chloride in the exhaust gases does not exceed 10 ppm, or equivalent as provided in § 61.66. This requirement does not apply to equipment that has been opened, is out of operation, and met the requirement in § 61.65(b)(6)(i) before being opened.

[51 FR 34909, Sept. 30, 1986]

§ 61.64 Emission standard for polyvinyl chloride plants.

An owner or operator of a polyvinyl chloride plant shall comply with the requirements of this section and § 61.65.

(a) *Reactor.* The following requirements apply to reactors:

(1) The concentration of vinyl chloride in each exhaust gas stream from each reactor is not to exceed 10 ppm (average for 3-hour period), except as provided in paragraph (a)(2) of this section and § 61.65(a).

(2) The reactor opening loss from each reactor is not to exceed 0.02 g vinyl chloride/kg (0.00002 lb vinyl chloride/lb) of polyvinyl chloride product, except as provided in paragraph (f)(1) of this section, with the product determined on a dry solids basis. This requirement does not apply to prepolymerization reactors in the bulk process. This requirement does apply to postpolymerization reactors in the bulk process, where the product means the gross product of prepolymerization and postpolymerization.

(3) Manual vent valve discharge. Except for an emergency manual vent valve discharge, there is to be no discharge to the atmosphere from any manual vent valve on a polyvinyl chloride reactor in vinyl chloride service. An emergency manual vent valve discharge means a discharge to the atmosphere which could not have been avoided by taking measures to prevent the discharge. Within 10 days of any discharge to the atmosphere from any manual vent valve, the owner or operator of the source from which the discharge occurs shall submit to the Administrator a report in writing containing information on the source, nature and cause of the discharge, the date and time of the discharge, the approximate total vinyl chloride loss during the discharge, the method used for determining the vinyl chloride loss (the calculation of the vinyl chloride loss), the action that was taken to prevent the discharge, and measures adopted to prevent future discharges.

(b) *Stripper*. The concentration of vinyl chloride in each exhaust gas stream from each stripper is not to exceed 10 ppm (average for 3-hour period), except as provided in § 61.65(a). This requirement does not apply to equipment that has been opened, is out of operation, and met the requirement § 61.65(b)(6)(i) before being opened.

(c) *Mixing, weighing, and holding containers*. The concentration of vinyl chloride in each exhaust gas stream from each mixing, weighing, or holding container in vinyl chloride service which precedes the stripper (or the reactor if the plant has no stripper) in the plant process flow is not to exceed 10 ppm (average for 3-hour period),

except as provided in § 61.65(a). This requirement does not apply to equipment that has been opened, is out of operation, and met the requirement in § 61.65(b)(6)(i) before being opened.

(d) *Monomer recovery system*. The concentration of vinyl chloride in each exhaust gas stream from each monomer recovery system is not to exceed 10 ppm (average for 3-hour period), except as provided in § 61.65(a). This requirement does not apply to equipment that has been opened, is out of operation, and met the requirement in § 61.65(b)(6)(i) before being opened.

(e) *Sources following the stripper(s)*. The following requirements apply to emissions of vinyl chloride to the atmosphere from the combination of all sources following the stripper(s) [or the reactor(s) if the plant has no stripper(s)] in the plant process flow including but not limited to, centrifuges, concentrators, blend tanks, filters, dryers, conveyor air discharges, baggers, storage containers, and in-process wastewater, except as provided in paragraph (f) of this section:

(1) In polyvinyl chloride plants using stripping technology to control vinyl chloride emissions, the weighted average residual vinyl chloride concentration in all grades of polyvinyl chloride resin processed through the stripping operation on each calendar day, measured immediately after the stripping operation is completed, may not exceed:

(i) 2000 ppm for polyvinyl chloride dispersion resins, excluding latex resins;

(ii) 400 ppm for all other polyvinyl chloride resins, including latex resins, averaged separately for each type of resin; or

(2) In polyvinyl chloride plants controlling vinyl chloride emissions with technology other than stripping or in addition to stripping, emissions of vinyl chloride to the atmosphere may not exceed:

(i) 2 g/kg (0.002 lb/lb) product from the stripper(s) [or reactor(s) if the plant has no stripper(s)] for dispersion polyvinyl chloride resins, excluding latex resins, with the product determined on a dry solids basis;

(ii) 0.4 g/kg (0.0004 lb/lb) product from the strippers for reactor(s) if the

plant has no stripper(s)] for all other polyvinyl chloride resins, including latex resins, with the product determined on a dry solids basis.

(3) The provisions of this paragraph apply at all times including when off-specification or other types of resins are made.

(f) Reactor used as stripper. When a nonbulk resin reactor is used as a stripper this paragraph may be applied in lieu of § 61.64 (a)(2) and (e)(1):

(1) The weighted average emissions of vinyl chloride from reactor opening loss and all sources following the reactor used as a stripper from all grades of polyvinyl chloride resin stripped in the reactor on each calendar day may not exceed:

(i) 2.02 g/kg (0.00202 lb/lb) of polyvinyl chloride product for dispersion polyvinyl chloride resins, excluding latex resins, with the product determined on a dry solids basis.

(ii) 0.42 g/kg (0.00042 lb/lb) of polyvinyl chloride product for all other polyvinyl chloride resins, including latex resins, with the product determined on a dry solids basis.

[41 FR 46564, Oct. 21, 1976, as amended at 51 FR 34909, Sept. 30, 1986; 53 FR 36972, Sept. 23, 1988]

§ 61.65 Emission standard for ethylene dichloride, vinyl chloride and polyvinyl chloride plants.

An owner or operator of an ethylene dichloride, vinyl chloride, and/or polyvinyl chloride plant shall comply with the requirements of this section.

(a) *Relief valve discharge.* Except for an emergency relief discharge, there is to be no discharge to the atmosphere from any relief valve on any equipment in vinyl chloride service. An emergency relief discharge means a discharge which could not have been avoided by taking measures to prevent the discharge. Within 10 days of any relief valve discharge, the owner or operator of the source from which the relief valve discharge occurs shall submit to the Administrator a report in writing containing information on the source, nature and cause of the discharge, the date and time of the discharge, the approximate total vinyl chloride loss during the discharge, the method used for determining the vinyl

chloride loss (the calculation of the vinyl chloride loss), the action that was taken to prevent the discharge, and measures adopted to prevent future discharges.

(b) *Fugitive emission sources—(1) Loading and unloading lines.* Vinyl chloride emissions from loading and unloading lines in vinyl chloride service which are opened to the atmosphere after each loading or unloading operation are to be minimized as follows:

(i) After each loading or unloading operation and before opening a loading or unloading line to the atmosphere, the quantity of vinyl chloride in all parts of each loading or unloading line that are to be opened to the atmosphere is to be reduced so that the parts combined contain no greater than $0.0038 \text{ m}^3 (0.13 \text{ ft}^3)$ of vinyl chloride, at standard temperature and pressure; and

(ii) Any vinyl chloride removed from a loading or unloading line in accordance with paragraph (b)(1)(i) of this section is to be ducted through a control system from which the concentration of vinyl chloride in the exhaust gases does not exceed 10 ppm (average for 3-hour period), or equivalent as provided in § 61.66.

(2) *Slip gauges.* During loading or unloading operations, the vinyl chloride emissions from each slip gauge in vinyl chloride service are to be minimized by ducting any vinyl chloride discharged from the slip gauge through a control system from which the concentration of vinyl chloride in the exhaust gases does not exceed 10 ppm (average for 3-hour period), or equivalent as provided in § 61.66.

(3) Leakage from pump, compressor, and agitator seals:

(i) *Rotating pumps.* Vinyl chloride emissions from seals on all rotating pumps in vinyl chloride service are to be minimized by installing sealless pumps, pumps with double mechanical seals or equivalent as provided in § 61.66. If double mechanical seals are used, vinyl chloride emissions from the seals are to be minimized by maintaining the pressure between the two seals so that any leak that occurs is into the pump; by ducting any vinyl chloride between the two seals

through a control system from which the concentration of vinyl chloride in the exhaust gases does not exceed 10 ppm; or equivalent as provided in § 61.66. Compliance with the provisions of 40 CFR part 61 subpart V demonstrates compliance with the provisions of this paragraph.

(ii) *Reciprocating pumps.* Vinyl chloride emissions from seals on all reciprocating pumps in vinyl chloride service are to be minimized by installing double outboard seals, or equivalent as provided in § 61.66. If double outboard seals are used, vinyl chloride emissions from the seals are to be minimized by maintaining the pressure between the two seals so that any leak that occurs is into the pump; by ducting any vinyl chloride between the two seals through a control system from which the concentration of vinyl chloride in the exhaust gases does not exceed 10 ppm; or equivalent as provided in § 61.66. Compliance with the provisions of 40 CFR part 61 subpart V demonstrates compliance with the provisions of this paragraph.

(iii) *Rotating compressor.* Vinyl chloride emissions from seals on all rotating compressors in vinyl chloride service are to be minimized by installing compressors with double mechanical seals, or equivalent as provided in § 61.66. If double mechanical seals are used, vinyl chloride emissions from the seals are to be minimized by maintaining the pressure between the two seals so that any leak that occurs is into the compressor; by ducting any vinyl chloride between the two seals through a control system from which the concentration of vinyl chloride in the exhaust gases does not exceed 10 ppm; or equivalent as provided in § 61.66. Compliance with the provisions of 40 CFR part 61 subpart V demonstrates compliance with the provisions of this paragraph.

(iv) *Reciprocating compressors.* Vinyl chloride emissions from seals on all reciprocating compressors in vinyl chloride service are to be minimized by installing double outboard seals, or equivalent as provided in § 61.66. If double outboard seals are used, vinyl chloride emissions from the seals are to be minimized by maintaining the pressure between the two seals so that

any leak that occurs is into the compressor; by ducting any vinyl chloride between the two seals through a control system from which concentration of vinyl chloride in the exhaust gases does not exceed 10 ppm; or equivalent as provided in § 61.66. Compliance with the provisions of 40 CFR part 61 subpart V demonstrates compliance with the provisions of this paragraph.

(v) *Agitator.* Vinyl chloride emissions from seals on all agitators in vinyl chloride service are to be minimized by installing agitators with double mechanical seals, or equivalent as provided in § 61.66. If double mechanical seals are used, vinyl chloride emissions from the seals are to be minimized by maintaining the pressure between the two seals so that any leak that occurs is into the agitated vessel; by ducting any vinyl chloride between the two seals through a control system from which the concentration of vinyl chloride in the exhaust gases does not exceed 10 ppm; or equivalent as provided in § 61.66.

(4) *Leaks from relief valves.* Vinyl chloride emissions due to leaks from each relief valve on equipment in vinyl chloride service shall comply with § 61.242-4 of subpart V of this part.

(5) *Manual venting of gases.* Except as provided in § 61.64(a)(3), all gases which are manually vented from equipment in vinyl chloride service are to be ducted through a control system from which the concentration of vinyl chloride in the exhaust gases does not exceed 10 ppm (average for 3-hour period); or equivalent as provided in § 61.66.

(6) *Opening of equipment.* Vinyl chloride emissions from opening of equipment (including prepolymerization reactors used in the manufacture of bulk resins and loading or unloading lines that are not opened to the atmosphere after each loading or unloading operation) are to be minimized as follows:

(i) Before opening any equipment for any reason, the quantity of vinyl chloride which is contained therein is to be reduced to an amount which occupies a volume of no more than 2.0 percent of the equipment's containment volume or 0.0950 cubic meters

(25 gallons), whichever is larger, at standard temperature and pressure.

(ii) Any vinyl chloride removed from the equipment in accordance with paragraph (b)(6)(i) of this section is to be ducted through a control system from which the concentration of vinyl chloride in the exhaust gases does not exceed 10 ppm (average for 3-hour period); or equivalent as provided in § 61.66.

(7) *Samples.* Unused portions of samples containing at least 10 percent by weight vinyl chloride are to be returned to the process or destroyed in a control device from which concentration of vinyl chloride in the exhaust gas does not exceed 10 ppm (average for 3-hour period) or equivalent as provided in § 61.66. Sampling techniques are to be such that sample containers in vinyl chloride service are purged into a closed process system. Compliance with the provisions of 40 CFR part 61 subpart V demonstrates compliance with the provisions of this paragraph.

(8) *Leak detection and elimination.* Vinyl chloride emissions due to leaks from equipment in vinyl chloride service are to be minimized as follows:

(i) A reliable and accurate vinyl chloride monitoring system shall be operated for detection of major leaks and identification of the general area of the plant where a leak is located. A vinyl chloride monitoring system means a device which obtains air samples from one or more points on a continuous sequential basis and analyzes the samples with gas chromatography or, if the owner or operator assumes that all hydrocarbons measured are vinyl chloride, with infrared spectrophotometry, flame ion detection, or an equivalent or alternative method. The vinyl chloride monitoring system shall be operated according to a program developed by the plant owner or operator. The owner or operator shall submit a description of the program to the Administrator within 45 days of the effective date of these regulations, unless a waiver of compliance is granted under § 61.11, or the program has been approved and the Administrator does not request a review of the program. Approval of a program will be

granted by the Administrator provided he finds:

(A) The location and number of points to be monitored and the frequency of monitoring provided for in the program are acceptable when they are compared with the number of pieces of equipment in vinyl chloride service and size and physical layout of the plant.

(B) It contains a definition of leak which is acceptable when compared with the background concentrations of vinyl chloride in the areas of the plant to be monitored by the vinyl chloride monitoring system. Measurements of background concentrations of vinyl chloride in the areas of the plant to be monitored by the vinyl chloride monitoring system are to be included with the description of the program. The definition of leak for a given plant may vary among the different areas within the plant and is also to change over time as background concentrations in the plant are reduced.

(C) It contains an acceptable plan of action to be taken when a leak is detected.

(D) It provides for an acceptable calibration and maintenance schedule for the vinyl chloride monitoring system and portable hydrocarbon detector. For the vinyl chloride monitoring system, a daily span check is to be conducted with a concentration of vinyl chloride equal to the concentration defined as a leak according to paragraph (b)(8)(i)(B) of this section. The calibration is to be done with either:

(1) A calibration gas mixture prepared from the gases specified in sections 5.2.1. and 5.2.2. of Test Method 106 and in accordance with section 7.1 of Test Method 106, or

(2) A calibration gas cylinder standard containing the appropriate concentration of vinyl chloride. The gas composition of the calibration gas cylinder standard is to have been certified by the manufacturer. The manufacturer must have recommended a maximum shelf life for each cylinder so that the concentration does not change greater than ± 5 percent from the certified value. The date of gas cylinder preparation, certified vinyl chloride concentration, and recom-

mended maximum self life must have been affixed to the cylinder before shipment from the manufacturer to the buyer. If a gas chromatograph is used as the vinyl chloride monitoring system, these gas mixtures may be directly used to prepare a chromatograph calibration curve as described in section 7.3 of Test Method 106. The requirements in section 5.2.3.1. and 5.2.3.2. of Test Method 106 for certification of cylinder standards and for establishment and verification of calibration standards are to be followed.

(ii) For each process unit subject to this subpart, a formal leak detection and repair program shall be implemented consistent with subpart V of this part, except as provided in paragraph (b)(8)(iii) of this section. This program is to be implemented within 90 days of the effective date of these regulations, unless a waiver of compliance is granted under § 61.11. Except as provided in paragraph (b)(8)(ii)(E) of this section, an owner or operator shall be exempt from § 61.242-1(d), § 61.242-7 (a), (b), and (c), § 61.246, and § 61.247 of subpart V of this part for any process unit in which the percentage of leaking valves is demonstrated to be less than 2.0 percent, as determined in accordance with the following:

(A) A performance test as specified in paragraph (b)(8)(ii)(B) of this section shall be conducted initially within 90 days of the effective date of these regulations, annually, and at times requested by the Administrator.

(B) For each performance test, a minimum of 200 or 90 percent, whichever is less, of the total valves in VOC service (as defined in § 60.481 of subpart VV of part 60) within the process unit shall be randomly selected and monitored within 1 week by the methods specified in § 61.245(b) of this part. If an instrument reading of 10,000 ppm or greater is measured, a leak is detected. The leak percentage shall be determined by dividing the number of valves in VOC service for which leaks are detected by the number of tested valves in VOC service.

(C) If a leak is detected, it shall be repaired in accordance with § 61.242-7 (d) and (e) of subpart V of this part.

(D) The results of the performance test shall be submitted in writing to the Administrator in the first quarterly report following the performance test as part of the reporting requirements of § 61.70.

(E) Any process unit in which the percentage of leaking valves is found to be greater than 2.0 percent according to the performance test prescribed in paragraph (b)(8)(ii)(B) of this section must comply with all provisions of subpart V of this part within 90 days.

(iii) Open-ended valves or lines located on multiple service process lines which operate in vinyl chloride service less than 10 percent of the time are exempt from the requirements of § 61.242-6 of subpart V, provided the open-ended valves or lines are addressed in the monitoring system required by paragraph (b)(8)(i) of this section. The Administrator may apply this exemption to other existing open-ended valves or lines that are demonstrated to require significant retrofit cost to comply with the requirements of § 61.242-6 of subpart V.

(9) *Inprocess wastewater.* Vinyl chloride emissions to the atmosphere from inprocess wastewater are to be reduced as follows:

(i) The concentration of vinyl chloride in each inprocess wastewater stream containing greater than 10 ppm vinyl chloride measured immediately as it leaves a piece of equipment and before being mixed with any other inprocess wastewater stream is to be reduced to no more than 10 ppm by weight before being mixed with any other inprocess wastewater stream which contains less than 10 ppm vinyl chloride; before being exposed to the atmosphere; before being discharged to a wastewater treatment process; or before being discharged untreated as a wastewater. This paragraph does apply to water which is used to displace vinyl chloride from equipment before it is opened to the atmosphere in accordance with § 61.64(a)(2) or paragraph (b)(6) of this section, but does not apply to water which is used to wash out equipment after the equipment has already been opened to the atmosphere in accordance with

§ 61.64(a)(2) or paragraph (b)(6) of this section.

(ii) Any vinyl chloride removed from the inprocess wastewater in accordance with paragraph (b)(9)(i) of this section is to be ducted through a control system from which the concentration of vinyl chloride in the exhaust gases does not exceed 10 ppm (average for 3-hour period); or equivalent as provided in § 61.66.

(c) The requirements in paragraphs (b)(1), (b)(2), (b)(5), (b)(6), (b)(7) and (b)(8) of this section are to be incorporated into a standard operating procedure, and made available upon request for inspection by the Administrator. The standard operating procedure is to include provisions for measuring the vinyl chloride in equipment 4.75 m³ (1255 gal) in volume for which an emission limit is prescribed in § 61.65(b)(6)(i) after opening the equipment and using Test Method 106, a portable hydrocarbon detector, or an alternative method. The method of measurement is to meet the requirements in § 61.67(g)(5)(i)(A) or (g)(5)(i)(B).

[41 FR 46564, Oct. 21, 1976; 41 FR 53017, Dec. 3, 1976, as amended at 42 FR 29006, June 7, 1977; 51 FR 34910, Sept. 30, 1986; 53 FR 36972, Sept. 23, 1988]

§ 61.66 Equivalent equipment and procedures.

Upon written application from an owner or operator, the Administrator may approve use of equipment or procedures which have been demonstrated to his satisfaction to be equivalent in terms of reducing vinyl chloride emissions to the atmosphere to those prescribed for compliance with a specific paragraph of this subpart.

[51 FR 34912, Sept. 30, 1986]

§ 61.67 Emission tests.

(a) Unless a waiver of emission testing is obtained under § 61.13, the owner or operator of a source to which this subpart applies shall test emissions from the source.

(1) Within 90 days of the effective date in the case of an existing source or a new source which has an initial startup date preceding the effective date, or

(2) Within 90 days of startup in the case of a new source, initial startup of which occurs after the effective date.

(b) The owner or operator shall provide the Administrator at least 30 days prior notice of an emission test to afford the Administrator the opportunity to have an observer present during the test.

(c) Any emission test is to be conducted while the equipment being tested is operating at the maximum production rate at which the equipment will be operated and under other relevant conditions as may be specified by the Administrator based on representative performance of the source.

(d) [Reserved]

(e) When at all possible, each sample is to be analyzed within 24 hours, but in no case in excess of 72 hours of sample collection. Vinyl chloride emissions are to be determined within 30 days after the emission test. The owner or operator shall report the determinations to the Administrator by a registered letter dispatched before the close of the next business day following the determination.

(f) The owner or operator shall retain at the plant and make available, upon request, for inspection by the Administrator, for a minimum of 3 years, records of emission test results and other data needed to determine emissions.

(g) Unless otherwise specified, the owner or operator shall use test Test Methods in Appendix B to this part for each test as required by paragraphs (g)(1), (g)(2), (g)(3), (g)(4), and (g)(5) of this section, unless an alternative method has been approved by the Administrator. If the Administrator finds reasonable grounds to dispute the results obtained by an alternative method, he may require the use of a reference method. If the results of the reference and alternative methods do not agree, the results obtained by the reference method prevail, and the Administrator may notify the owner or operator that approval of the method previously considered to be alternative is withdrawn. Whenever Test Method 107 is specified, and the conditions in Section 1.1, "Applicability" of Method 107A are met, Method 107A may be used.

(1) Test Method 106 is to be used to determine the vinyl chloride emissions from any source for which an emission limit is prescribed in § 61.62 (a) or (b) § 61.63(a), or § 61.64(a)(1), (b), (c), or (d), or from any control system to which reactor emissions are required to be ducted in § 61.64(a)(2) or to which fugitive emissions are required to be ducted is § 61.65(b)(1)(ii), (b)(2), (b)(5), (b)(6)(ii), or (b)(9)(ii).

(i) For each run, one sample is to be collected. The sampling site is to be at least two stack or duct diameters downstream and one half diameter upstream from any flow disturbance such as a bend, expansion, contraction, or visible flame. For a rectangular cross section an equivalent diameter is to be determined from the following equation:

$$\text{equivalent diameter} = 2 \frac{(\text{length}) (\text{width})}{\text{length} + \text{width}}$$

The sampling point in the duct is to be at the centroid of the cross section. The sample is to be extracted at a rate proportional to the gas velocity at the sampling point. The sample is to contain a minimum volume of 50 liters corrected to standard conditions and is to be taken over a period as close to 1 hour as practicable.

(ii) Each emission test is to consist of three runs. For the purpose of determining emissions, the average of results of all runs is to apply. The average is to be computed on a time weighted basis.

(iii) For gas streams containing more than 10 percent oxygen the concentration of vinyl chloride as determined by Test Method 106 is to be corrected to 10 percent oxygen (dry basis) for determination of emissions by using the following equation:

$$C_b (\text{corrected}) = C_b \frac{10.9}{20.9} - \text{percent } O_2$$

where:

$C_b (\text{corrected})$ = The concentration of vinyl chloride in the exhaust gases, corrected to 10-percent oxygen.

C_b = The concentration of vinyl chloride as measured by Test Method 106.

20.9 = Percent oxygen in the ambient air at standard conditions.

10.9 = Percent oxygen in the ambient air at standard conditions, minus the 10.0-percent oxygen to which the correction is being made.

Percent O_2 = Percent oxygen in the exhaust gas as measured by Reference Method 3 in Appendix A of part 60 of this chapter.

(iv) For those emission sources where the emission limit is prescribed in terms of mass rather than concentration, mass emissions in kg/100 kg product are to be determined by using the following equation:

$$C_{sx} = [C_b (2.60) Q 10^{-6}] [100] / Z$$

where:

C_{sx} = kg vinyl chloride/100 kg product.

C_b = The concentration of vinyl chloride as measured by Test Method 106.

2.60 = Density of vinyl chloride at one atmosphere and 20° C in kg/m³

Q = Volumetric flow rate in m³/hr as determined by Reference Method 2 of Appendix A to part 60 of this chapter.

10⁻⁶ = Conversion factor for ppm.

Z = Production rate (kg/hr).

(2) Test Method 107 or Method 601 (incorporated by reference as specified in § 61.18) is to be used to determine the concentration of vinyl chloride in each inprocess wastewater stream for which an emission limit is prescribed in § 61.65(b)(9)(i).

(3) When a stripping operation is used to attain the emission limits in § 61.64 (e) and (f), emissions are to be determined using Test Method 107 as follows:

(i) The number of strippers (or reactors used as strippers) and samples and the types and grades of resin to be sampled are to be determined by the Administrator for each individual plant at the time of the test based on the plant's operation.

(ii) Each sample is to be taken immediately following the stripping operation.

(iii) The corresponding quantity of material processed by each stripper (or reactor used as a stripper) is to be determined on a dry solids basis and by a method submitted to and approved by the Administrator.

(iv) At the prior request of the Administrator, the owner or operator shall provide duplicates of the samples required in paragraph (g)(3)(i) of this section.

(4) Where control technology other than or in addition to a stripping operation is used to attain the emission

limit in § 61.64(e), emissions are to be determined as follows:

(i) Test Method 106 is to be used to determine atmospheric emissions from all of the process equipment simultaneously. The requirements of paragraph (g)(1) of this section are to be met.

(ii) Test Method 107 is to be used to determine the concentration of vinyl chloride in each inprocess wastewater stream subject to the emission limit prescribed in § 61.64(e). The mass of vinyl chloride in kg/100 kg product in each inprocess wastewater stream is to be determined by using the following equation:

$$C_{ax} = [C_d R 10^{-6}] [100]/Z$$

where:

C_{ax} = kg vinyl chloride/100 kg product.

C_d = the concentration of vinyl chloride as measured by Test Method 107.

R = water flow rate in l/hr, determined in accordance with a method which has been submitted to and approved by the Administrator.

10^{-6} = Conversion factor for ppm.

Z = Production rate (kg/hr), determined in accordance with a method which has been submitted and approved by the Administrator.

(5) The reactor opening loss for which an emission limit is prescribed in § 61.64(a)(2) is to be determined. The number of reactors for which the determination is to be made is to be specified by the Administrator for each individual plant at the time of the determination based on the plant's operation.

(i) Except as provided in paragraph (g)(5)(ii) of this section, the reactor opening loss is to be determined using the following equation:

$$C = W (2.60) (10^{-6}) (C_b) / YZ$$

where:

C = kg vinyl chloride emissions/kg product.

W = Capacity of the reactor in m^3 .

2.60 = Density of vinyl chloride at one atmosphere and $20^\circ C$ in kg/m^3 .

10^{-6} = Conversion factor for ppm.

C_b = ppm by volume vinyl chloride as determined by Test Method 106 or a portable hydrocarbon detector which measures hydrocarbons with a sensitivity of at least 10 ppm.

Y = Number of batches since the reactor was last opened to the atmosphere.

Z = Average kg of polyvinyl chloride produced per batch in the number of batches since the reactor was last opened to the atmosphere.

(A) If Method 106 is used to determine the concentration of vinyl chloride (C_b), the sample is to be withdrawn at a constant rate with a probe of sufficient length to reach the vessel bottom from the manhole. Samples are to be taken for 5 minutes within 6 inches of the vessel bottom, 5 minutes near the vessel center, and 5 minutes near the vessel top.

(B) If a portable hydrocarbon detector is used to determine the concentration of vinyl chloride (C_b), a probe of sufficient length to reach the vessel bottom from the manhole is to be used to make the measurements. One measurement will be made within 6 inches of the vessel bottom, one near the vessel center and one near the vessel top. Measurements are to be made at each location until the reading is stabilized. All hydrocarbons measured are to be assumed to be vinyl chloride.

(C) The production rate of polyvinyl chloride (Z) is to be determined by a method submitted to and approved by the Administrator.

(ii) A calculation based on the number of evacuations, the vacuum involved, and the volume of gas in the reactor is hereby approved by the Administrator as an alternative method for determining reactor opening loss for postpolymerization reactors in the manufacture of bulk resins. Calculation methods based on techniques other than repeated evacuation of the reactor may be approved by the Administrator for determining reactor opening loss for postpolymerization reactors in the manufacture of bulk resins.

(6) For a reactor that is used as a stripper, the emissions of vinyl chloride from reactor opening loss and all sources following the reactor used as a stripper for which an emission limit is prescribed in § 61.64(f) are to be determined. The number of reactors for which the determination is to be made is to be specified by the Administrator for each individual plant at the time of the determination based on the plant's operation.

(i) For each batch stripped in the reactor, the following measurements are to be made:

(A) The concentration (ppm) of vinyl chloride in resin after stripping, measured according to paragraph (g)(3) of this section;

(B) The reactor vacuum (mm Hg) at end of strip from plant instrument; and

(C) The reactor temperature (°C) at end of strip from plant instrument.

(ii) For each batch stripped in the reactor, the following information is to be determined:

(A) The vapor pressure (mm Hg) of water in the reactor at end of strip from the following table:

Reactor vapor temperature (°C)	H ₂ O vapor pressure (mm Hg)	Reactor temperature (°C)	H ₂ O vapor pressure (mm Hg)	Reactor temperature (°C)	H ₂ O vapor pressure (mm Hg)
40	55.3	62	163.8	84	416.8
41	58.3	63	171.4	85	433.6
42	61.5	64	179.3	86	450.9
43	64.8	65	187.5	87	468.7
44	68.3	66	196.1	88	487.1
45	71.9	67	205.0	89	506.1
46	75.6	68	214.2	90	525.8
47	79.6	69	223.7	91	546.0
48	83.7	70	233.7	92	567.0
49	88.0	71	243.9	93	588.6
50	92.5	72	254.6	94	610.9
51	97.2	73	265.7	95	633.9
52	102.1	74	277.2	96	657.6
53	107.2	75	289.1	97	682.1
54	112.5	76	301.4	98	707.3
55	118.0	77	314.1	99	733.2
56	123.8	78	327.3	100	760.0
57	129.8	79	341.0		
58	136.1	80	355.1		
59	142.6	81	369.7		
60	149.4	82	384.9		

Reactor vapor temperature (°C)	H ₂ O vapor pressure (mm Hg)	Reactor temperature (°C)	H ₂ O vapor pressure (mm Hg)	Reactor temperature (°C)	H ₂ O vapor pressure (mm Hg)
61	156.4	83	400.6		

(B) The partial pressure (mm Hg) of vinyl chloride in reactor at end of strip from the following equation:

$$PPVC = 760 - RV - VPW$$

where:

PPVC = partial pressure of vinyl chloride, in mm Hg

760 = atmospheric pressure at 0 °C, in mm Hg

RV = absolute value of reactor vacuum, in mm Hg

VPW = vapor pressure of water, in mm Hg

(C) The reactor vapor space volume (m³) at end of strip from the following equation:

$$RVS\bar{V} = RC - WV - \frac{PVCW}{1,400}$$

where:

RVS $\bar{V}$ = reactor vapor space volume, in m³

RC = reactor capacity, in m³

WV = volume of water in reactor from recipe, in m³

PVCW = dry weight of polyvinyl chloride in reactor from recipe, in kg

1,400 = typical density of polyvinyl chloride, in kg/m³

(iii) For each batch stripped in the reactor, the combined reactor opening loss and emissions from all sources following the reactor used as a stripper is to be determined using the following equation:

$$C = (PPMVC)(10^{-3}) + \frac{(PPVC)(RVS\bar{V})(1,002)}{(PVCW)(273 + RT)}$$

where:

C = g vinyl chloride/kg polyvinyl chloride product

PPMVC = concentration of vinyl chloride in resin after stripping, in ppm

10⁻³ = conversion factor for ppm

PPVC = partial pressure of vinyl chloride determined according to paragraph (g)(6)(ii)(B) of this section, in mm Hg

RVS $\bar{V}$ = reactor vapor space volume determined according to paragraph (g)(6)(ii)(C) of this section, in m³

1,002 = ideal gas constant in g · °K/mm Hg · m³ for vinyl chloride

PVCW = dry weight of polyvinyl chloride in reactor from recipe, in kg

273 = conversion factor for °C to °K

RT = reactor temperature, in °C

(h)(1) Each piece of equipment within a process unit that can reasonably contain equipment in vinyl chloride service is presumed to be in vinyl chloride service unless an owner or operator demonstrates that the piece of equipment is not in vinyl chloride service. For a piece of equipment to be considered not in vinyl chloride service, it must be determined that the percent vinyl chloride content can be reasonably expected not to exceed 10 percent by weight for liquid streams or contained liquid volumes and 10 percent by volume for gas streams or contained gas volumes, which also includes gas volumes above liquid streams or contained liquid volumes. For purposes of determining the percent vinyl chloride content of the process fluid that is contained in or contacts equipment, procedures that conform to the methods described in ASTM Method D-2267 (incorporated by reference as specified in § 61.18) shall be used.

(2)(i) An owner or operator may use engineering judgment rather than the procedures in paragraph (h)(1) of this section to demonstrate that the percent vinyl chloride content does not exceed 10 percent by weight for liquid streams and 10 percent by volume for gas streams, provided that the engineering judgment demonstrates that the vinyl chloride content clearly does not exceed 10 percent. When an owner or operator and the Administrator do not agree on whether a piece of equipment is not in vinyl chloride service, however, the procedures in paragraph (h)(1) of this section shall be used to resolve the disagreement.

(ii) If an owner or operator determines that a piece of equipment is in vinyl chloride service, the determination can be revised only after following the procedures in paragraph (h)(1) of this section.

(3) Samples used in determining the percent vinyl chloride content shall be representative of the process fluid that is contained in or contacts the equipment.

[41 FR 46564, Oct. 21, 1976, as amended at 42 FR 29007, June 7, 1977; 47 FR 39486, Sept. 8, 1982; 50 FR 46295, Nov. 7, 1985; 51 FR 34912, Sept. 30, 1986]

§ 61.68 Emission monitoring.

(a) A vinyl chloride monitoring system is to be used to monitor on a continuous basis the emissions from the sources for which emission limits are prescribed in §§ 61.62 (a) and (b), 61.63(a), and 61.64 (a)(1), (b), (c), and (d), and for any control system to which reactor emissions are required to be ducted in § 61.64(a)(2) or to which fugitive emissions are required to be ducted in § 61.65 (b)(1)(ii), and (b)(2), (b)(5), (b)(6) (ii), and (b)(9)(ii).

(b) The vinyl chloride monitoring system(s) used to meet the requirement in paragraph (a) of this section is to be a device which obtains air samples from one or more points on a continuous sequential basis and analyzes the samples with gas chromatography or, if the owner or operator assumes that all hydrocarbons measured are vinyl chloride, with infrared spectrophotometry, flame ion detection, or an alternative method. The vinyl chloride monitoring system used to meet the requirements in § 61.65(b)(8)(i) may be used to meet the requirements of this section.

(c) A daily span check is to be conducted for each vinyl chloride monitoring system used. For all of the emission sources listed in paragraph (a) of this section, except the one for which an emission limit is prescribed in § 61.62(b), the daily span check is to be conducted with a concentration of vinyl chloride equal to 10 ppm. For the emission source for which an emission limit is prescribed in § 61.62(b), the daily span check is to be conducted with a concentration of vinyl chloride which is determined to be equivalent to the emission limit for that source based on the emission test required by § 61.67. The calibration is to be done with either:

(1) A calibration gas mixture prepared from the gases specified in sections 5.2.1 and 5.2.2 of Test Method 106 and in accordance with section 7.1 of Test Method 106, or

(2) A calibration gas cylinder standard containing the appropriate concentration of vinyl chloride. The gas composition of the calibration gas cylinder standard is to have been certified by the manufacturer. The manu-

facturer must have recommended a maximum shelf life for each cylinder so that the concentration does not change greater than ± 5 percent from the certified value. The date of gas cylinder preparation, certified vinyl chloride concentration and recommended maximum shelf life must have been affixed to the cylinder before shipment from the manufacturer to the buyer. If a gas chromatograph is used as the vinyl chloride monitoring system, these gas mixtures may be directly used to prepare a chromatograph calibration curve as described in section 7.3 of Test Method 106. The requirements in sections 5.2.3.1 and 5.2.3.2 of Test Method 106 for certification of cylinder standards and for establishment and verification of calibration standards are to be followed.

(d) When exhaust gas(es), having emission limits that are subject to the requirement of paragraph (a) of this section, are emitted to the atmosphere without passing through the control system and required vinyl chloride monitoring system, the vinyl chloride content of the emission shall be calculated (in units of each applicable emission limit) by best practical engineering judgment based on the discharge duration and known VC concentrations in the affected equipment as determined in accordance with § 61.67(h) or other acceptable method.

(e) For each 3-hour period, the vinyl chloride content of emissions subject to the requirements of paragraphs (a) and (d) of this section shall be averaged (weighted according to the proportion of time that emissions were continuously monitored and that emissions bypassed the continuous monitor) for purposes of reporting excess emissions under § 61.70(c)(1).

(f) For each vinyl chloride emission to the atmosphere determined in accordance with paragraph (e) of this section to be in excess of the applicable emission limits, the owner or operator shall record the identity of the source(s), the date, time, and duration of the excess emission, the cause of the excess emission, and the approximate total vinyl chloride loss during the excess emission, and the method used for determining the vinyl chloride loss. This information shall be re-

tained and made available for inspection by the Administrator as required by § 61.71(a).

[41 FR 46564, Oct. 21, 1976; 41 FR 53017, Dec. 3, 1976, as amended at 42 FR 29007, June 7, 1977; 50 FR 46295, Nov. 7, 1985; 51 FR 34913, Sept. 30, 1986]

§ 61.69 Initial report.

(a) An owner or operator of any source to which this subpart applies shall submit a statement in writing notifying the Administrator that the equipment and procedural specifications in § 61.65 (b)(1), (b)(2), (b)(3), (b)(4), (b)(5), (b)(6), (b)(7), and (b)(8) are being implemented.

(b)(1) In the case of an existing source or a new source which has an initial startup date preceding the effective date, the statement is to be submitted within 90 days of the effective date, unless a waiver of compliance is granted under § 61.11, along with the information required under § 61.10. If a waiver of compliance is granted, the statement is to be submitted on a date scheduled by the Administrator.

(2) In the case of a new source which did not have an initial startup date preceding the effective date, the statement is to be submitted within 90 days of the initial startup date.

(c) The statement is to contain the following information:

(1) A list of the equipment installed for compliance,

(2) A description of the physical and functional characteristics of each piece of equipment,

(3) A description of the methods which have been incorporated into the standard operating procedures for measuring or calculating the emissions for which emission limits are prescribed in § 61.65 (b)(1)(i) and (b)(6)(i),

(4) A statement that each piece of equipment is installed and that each piece of equipment and each procedure is being used.

§ 61.70 Reporting.

(a)(1) The owner or operator of any source to which this subpart applies shall submit to the Administrator on March 15, June 15, September 15, and December 15 of each year a report in

writing containing the information required by this section. The first report is to be submitted following the first full 3-month reporting period after the initial report is submitted.

(2) In the case of an existing source, the approved reporting schedule shall be used. In addition, quarterly reports shall be submitted exactly 3 months following the current reporting dates.

(b)(1) In the case of an existing source or a new source which has an initial startup date preceding the effective date, the first report is to be submitted within 180 days of the effective date, unless a waiver of compliance is granted under § 61.11. If a waiver of compliance is granted, the first report is to be submitted on a date scheduled by the Administrator.

(2) In the case of a new source which did not have an initial startup date preceding the effective date, the first report is to be submitted within 180 days of the initial startup date.

(c) Unless otherwise specified, the owner or operator shall use the Test Methods in Appendix B to this part to conduct emission tests as required by paragraphs (c)(2) and (c)(3) of this section, unless an alternative method has been approved by the Administrator. If the Administrator finds reasonable grounds to dispute the results obtained by an alternative method, he may require the use of a reference method. If the results of the reference and alternative methods do not agree, the results obtained by the reference method prevail, and the Administrator may notify the owner or operator that approval of the method previously considered to be alternative is withdrawn.

(1) The owner or operator shall include in the report a record of the vinyl chloride content of emissions for each 3-hour period during which average emissions are in excess of the emission limits in § 61.62 (a) or (b), § 61.63 (a), or § 61.64 (a)(1), (b), (c), or (d), or during which average emissions are in excess of the emission limits specified for any control system to which reactor emissions are required to be ducted in § 61.64 (a)(2) or to which fugitive emissions are required to be ducted in § 61.65 (b)(i)(ii), (b)(2), (b)(5), (b)(6)(ii), or (b)(9)(ii). The

number of 3-hour periods for which average emissions were determined during the reporting period shall be reported. If emissions in excess of the emission limits are not detected, the report shall contain a statement that no excess emissions have been detected. The emissions are to be determined in accordance with § 61.68(e).

(2) In polyvinyl chloride plants for which a stripping operation is used to attain the emission level prescribed in § 61.64(e), the owner or operator shall include in the report a record of the vinyl chloride content in the polyvinyl chloride resin.

(i) If batch stripping is used, one representative sample of polyvinyl chloride resin is to be taken from each batch of each grade of resin immediately following the completion of the stripping operation, and identified by resin type and grade and the date and time the batch is completed. The corresponding quantity of material processed in each stripper batch is to be recorded and identified by resin type and grade and the date and time the batch is completed.

(ii) If continuous stripping is used, one representative sample of polyvinyl chloride resin is to be taken for each grade of resin processed or at intervals of 8 hours for each grade of resin which is being processed, whichever is more frequent. The sample is to be taken as the resin flows out of the stripper and identified by resin type and grade and the date and time the sample was taken. The corresponding quantity of material processed by each stripper over the time period represented by the sample during the 8-hour period, is to be recorded and identified by resin type and grade and the date and time it represents.

(iii) The vinyl chloride content in each sample is to be determined by Test Method 107 as prescribed in § 61.67(g)(3).

(iv) [Reserved]

(v) The report to the Administrator by the owner or operator is to include a record of any 24-hour average resin vinyl chloride concentration, as determined in this paragraph, in excess of the limits prescribed in § 61.64(e). The vinyl chloride content found in each sample required by paragraphs

(c)(2)(i) and (c)(2)(ii) of this section shall be averaged separately for each type of resin, over each calendar day and weighted according to the quanti-

ty of each grade of resin processed by the stripper(s) that calendar day, according to the following equation:

$$A_T = \frac{\sum_{i=1}^n P_{Gi} M_{Gi}}{Q_T} = \frac{P_{G1} M_{G1} + P_{G2} M_{G2} + \dots + P_{Gn} M_{Gn}}{Q_T}$$

where:

A_T = 24-hour average concentration of type T resin in ppm (dry weight basis)

Q_T = Total production of type T resin over the 24-hour period, in kg.

T = Type of resin.

M_{Gi} = Concentration of vinyl chloride in one sample of grade G_i resin in ppm.

P_{Gi} = Production of grade G_i resin represented by the sample, in kg.

G_i = Grade of resin: e.g., G_1 , G_2 , G_3 .

n = Total number of grades of resin produced during the 24-hour period.

The number of 24-hour average concentrations for each resin type determined during the reporting period shall be reported. If no 24-hour average resin vinyl chloride concentrations in excess of the limits prescribed in § 61.64(e) are measured, the report shall state that no excess resin vinyl chloride concentrations were measured.

(vi) The owner or operator shall retain at the source and make available for inspection by the Administrator for a minimum of 3 years records of all data needed to furnish the information required by paragraph (c)(2)(v) of this section. The records are to contain the following information:

(A) The vinyl chloride content found in all the samples required in paragraphs (c)(2)(i) and (c)(2)(ii) of this section, identified by the resin type and grade and the time and date of the sample, and

(B) The corresponding quantity of polyvinyl chloride resin processed by the stripper(s), identified by the resin type and grade and the time and date it represents.

(3) The owner or operator shall include in the report a record of any emissions from each reactor opening in excess of the emission limits prescribed in § 61.64(a)(2). Emissions are

to be determined in accordance with § 61.67(g)(5), except that emissions for each reactor are to be determined. The number of reactor openings during the reporting period shall be reported. If emissions in excess of the emission limits are not detected, the report shall include a statement that excess emissions have not been detected.

(4) In polyvinyl chloride plants for which stripping in the reactor is used to attain the emission level prescribed in § 61.64(f), the owner or operator shall include in the report a record of the vinyl chloride emissions from reactor opening loss and all sources following the reactor used as a stripper.

(i) One representative sample of polyvinyl chloride resin is to be taken from each batch of each grade of resin immediately following the completion of the stripping operation, and identified by resin type and grade and the date and time the batch is completed. The corresponding quantity of material processed in each stripper batch is to be recorded and identified by resin type and grade and the date and time the batch is completed.

(ii) The vinyl chloride content in each sample is to be determined by Test Method 107 as prescribed in § 61.67(g)(3).

(iii) The combined emissions from reactor opening loss and all sources following the reactor used as a stripper are to be determined for each batch stripped in a reactor according to the procedure prescribed in § 61.67(g)(6).

(iv) The report to the Administrator by the owner or operator is to include a record of any 24-hour average combined reactor opening loss and emissions from all sources following the reactor used as a stripper as determined

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in this paragraph, in excess of the limits prescribed in § 61.64(f). The combined reactor opening loss and emissions from all sources following the reactor used as a stripper associated with each batch are to be averaged separately for each type of resin, over

each calendar day and weighted according to the quantity of each grade of resin stripped in reactors that calendar day as follows:

For each type of resin (suspension, dispersion, latex, bulk, other), the following calculation is to be performed:

$$A_T = \frac{\sum_{i=1}^n P_{Gi} C_{Gi}}{Q_T} = \frac{P_{G1} C_{G1} + P_{G2} C_{G2} + \dots + P_{Gn} C_{Gn}}{Q_T}$$

where:

A_T = 24-hour average combined reactor opening loss and emissions from all sources following the reactor used as a stripper, in g vinyl chloride/kg product (dry weight basis).

Q_T = Total production of resin in batches for which stripping is completed during the 24-hour period, in kg.

T = Type of resin.

C_{Gi} = Average combined reactor opening loss and emissions from all sources following the reactor used as a stripper of all batches of grade G_i resin for which stripping is completed during the 24-hour period, in g vinyl chloride/kg product (dry weight basis) (determined according to procedure prescribed in § 61.67(g)(6)).

P_{Gi} = Production of grade G_i resin in the batches for which C is determined, in kg.

G_i = Grade of resin e.g., G_1 , G_2 , and G_3 .

n = Total number of grades of resin in batches for which stripping is completed during the 24-hour period.

The number of 24-hour average emissions determined during the reporting period shall be reported. If no 24-hour average combined reactor opening loss and emissions from all sources following the reactor used as a stripper in excess of the limits prescribed in § 61.64(f) are determined, the report shall state that no excess vinyl chloride emissions were determined.

[41 FR 46564, Oct. 21, 1976 as amended at 42 FR 29007, June 7, 1977; 50 FR 46295, Nov. 7, 1985; 51 FR 34914, Sept. 30, 1986; 53 FR 36972, Sept. 23, 1988; 53 FR 46976, Nov. 21, 1988]

§ 61.71 Recordkeeping.

(a) The owner or operator of any source to which this subpart applies

shall retain the following information at the source and make it available for inspection to the Administrator for a minimum of 3 years:

(1) A record of the leaks detected by the vinyl chloride monitoring system, as required by § 61.65(b)(8), including the concentrations of vinyl chloride measured, analyzed, and recorded by the vinyl chloride detector, the location of each measurement and the date and approximate time of each measurement.

(2) A record of the leaks detected during routine monitoring with the portable hydrocarbon detector and the action taken to repair the leaks, as required by § 61.65(b)(8), including a brief statement explaining the location and cause of each leak detected with the portable hydrocarbon detector, the date and time of the leak, and any action taken to eliminate that leak.

(3) A record of emissions measured in accordance with § 61.68.

(4) A daily operating record for each polyvinyl chloride reactor, including pressures and temperatures.

[41 FR 46594, Oct. 21, 1976, as amended at 42 FR 29007, June 7, 1977; 51 FR 34914, Sept. 30, 1986]

Subpart G—[Reserved]

Subpart H—National Emission Standards for Emissions of Radionuclides Other Than Radon From Department of Energy Facilities

SOURCE: 54 FR 51695, Dec. 15, 1989, unless otherwise noted.

§ 61.90 Designation of facilities.

The provisions of this subpart apply to operations at any facility owned or operated by the Department of Energy that emits any radionuclide other than radon-222 and radon-220 into the air, except that this subpart does not apply to disposal at facilities subject to 40 CFR part 191, subpart B or 40 CFR part 192.

§ 61.91 Definitions.

As used in this subpart, all terms not defined here have the meaning given them in the Clean Air Act or 40 CFR part 61, subpart A. The following terms shall have the following specific meanings:

(a) *Effective dose equivalent* means the sum of the products of absorbed dose and appropriate factors to account for differences in biological effectiveness due to the quality of radiation and its distribution in the body of reference man. The unit of the effective dose equivalent is the rem. For purposes of this subpart, doses caused by radon-222 and its respective decay products formed after the radon is released from the facility are not included. The method for calculating effective dose equivalent and the definition of reference man are outlined in the International Commission on Radiological Protection's Publication No. 26.

(b) *Facility* means all buildings, structures and operations on one contiguous site.

(c) *Radionuclide* means a type of atom which spontaneously undergoes radioactive decay.

(d) *Residence* means any home, house, apartment building, or other place of dwelling which is occupied during any portion of the relevant year.

§ 61.92 Standard.

Emissions of radionuclides to the ambient air from Department of Energy facilities shall not exceed those amounts that would cause any member of the public to receive in any year an effective dose equivalent of 10 mrem/yr.

§ 61.93 Emission monitoring and test procedures.

(a) To determine compliance with the standard, radionuclide emissions shall be determined and effective dose equivalent values to members of the public calculated using EPA approved sampling procedures, computer models CAP-88 or AIRDOS-PC, or other procedures for which EPA has granted prior approval. DOE facilities for which the maximally exposed individual lives within 3 kilometers of all sources of emissions in the facility, may use EPA's COMPLY model and associated procedures for determining dose for purposes of compliance.

(b) Radionuclide emission rates from point sources (stacks or vents) shall be measured in accordance with the following requirements or other procedures for which EPA has granted prior approval:

(1) Effluent flow rate measurements shall be made using the following methods:

(i) Reference Method 2 of Appendix A to part 60 shall be used to determine velocity and volumetric flow rates for stacks and large vents.

(ii) Reference Method 2A of Appendix A to part 60 shall be used to measure flow rates through pipes and small vents.

(iii) The frequency of the flow rate measurements shall depend upon the variability of the effluent flow rate. For variable flow rates, continuous or frequent flow rate measurements shall be made. For relatively constant flow rates only periodic measurements are necessary.

(2) Radionuclides shall be directly monitored or extracted, collected and measured using the following methods:

(i) Reference Method 1 of Appendix A part 60 shall be used to select monitoring or sampling sites.

(ii) The effluent stream shall be directly monitored continuously with an in-line detector or representative samples of the effluent stream shall be withdrawn continuously from the sampling site following the guidance presented in ANSIN13.1-1969 "Guide to Sampling Airborne Radioactive Materials in Nuclear Facilities" (including

the guidance presented in Appendix A of ANSIN13.1) (incorporated by reference—see § 61.18). The requirements for continuous sampling are applicable to batch processes when the unit is in operation. Periodic sampling (grab samples) may be used only with EPA's prior approval. Such approval may be granted in cases where continuous sampling is not practical and radionuclide emission rates are relatively constant. In such cases, grab samples shall be collected with sufficient frequency so as to provide a representative sample of the emissions.

(iii) Radionuclides shall be collected and measured using procedures based on the principles of measurement described in Appendix B, Method 114. Use of methods based on principles of measurement different from those described in Appendix B, Method 114 must have prior approval from the Administrator. EPA reserves the right to approve measurement procedures.

(iv) A quality assurance program shall be conducted that meets the performance requirements described in Appendix B, Method 114.

(3) When it is impractical to measure the effluent flow rate at an existing source in accordance with the requirements of paragraph (b)(1) of this section or to monitor or sample an effluent stream at an existing source in accordance with the site selection and sample extraction requirements of paragraph (b)(2) of this section, the facility owner or operator may use alternative effluent flow rate measurement procedures or site selection and sample extraction procedures provided that:

(i) It can be shown that the requirements of paragraph (b) (1) or (2) of this section are impractical for the effluent stream.

(ii) The alternative procedure will not significantly underestimate the emissions.

(iii) The alternative procedure is fully documented.

(iv) The owner or operator has received prior approval from EPA.

(4)(i) Radionuclide emission measurements in conformance with the requirements of paragraph (b) of this section shall be made at all release points which have a potential to dis-

charge radionuclides into the air in quantities which could cause an effective dose equivalent in excess of 1% of the standard. All radionuclides which could contribute greater than 10% of the potential effective dose equivalent for a release point shall be measured. With prior EPA approval, DOE may determine these emissions through alternative procedures. For other release points which have a potential to release radionuclides into the air, periodic confirmatory measurements shall be made to verify the low emissions.

(ii) To determine whether a release point is subject to the emission measurement requirements of paragraph (b) of this section, it is necessary to evaluate the potential for radionuclide emissions for that release point. In evaluating the potential of a release point to discharge radionuclides into the air for the purposes of this section, the estimated radionuclide release rates shall be based on the discharge of the effluent stream that would result if all pollution control equipment did not exist, but the facilities operations were otherwise normal.

(5) Environmental measurements of radionuclide air concentrations at critical receptor locations may be used as an alternative to air dispersion calculations in demonstrating compliance with the standard if the owner or operator meets the following criteria:

(i) The air at the point of measurement shall be continuously sampled for collection of radionuclides.

(ii) Those radionuclides released from the facility, which are the major contributors to the effective dose equivalent must be collected and measured as part of the environmental measurement program.

(iii) Radionuclide concentrations which would cause an effective dose equivalent of 10% of the standard shall be readily detectable and distinguishable from background.

(iv) Net measured radionuclide concentrations shall be compared to the concentration levels in Table 2 of Appendix E to determine compliance with the standard. In the case of multiple radionuclides being released from a facility, compliance shall be demonstrated if the value for all radionuclides is less than the concentration

level in Table 2, and the sum of the fractions that result when each measured concentration value is divided by the value in Table 2 for each radionuclide is less than 1.

(v) A quality assurance program shall be conducted that meets the performance requirements described in Appendix B, Method 114.

(vi) Use of environmental measurements to demonstrate compliance with the standard is subject to prior approval of EPA. Applications for approval shall include a detailed description of the sampling and analytical methodology and show how the above criteria will be met.

§ 61.94 Compliance and reporting.

(a) Compliance with this standard shall be determined by calculating the highest effective dose equivalent to any member of the public at any off-site point where there is a residence, school, business or office. The owners or operators of each facility shall submit an annual report to both EPA headquarters and the appropriate regional office by June 30 which includes the results of the monitoring as recorded in DOE's Effluent Information System and the dose calculations required by § 61.93(a) for the previous calendar year.

(b) In addition to the requirements of paragraph (a) of this section, an annual report shall include the following information:

(1) The name and location of the facility.

(2) A list of the radioactive materials used at the facility.

(3) A description of the handling and processing that the radioactive materials undergo at the facility.

(4) A list of the stacks or vents or other points where radioactive materials are released to the atmosphere.

(5) A description of the effluent controls that are used on each stack, vent, or other release point and an estimate of the efficiency of each control device.

(6) Distances from the points of release to the nearest residence, school, business or office and the nearest farms producing vegetables, milk, and meat.

(7) The values used for all other user-supplied input parameters for the computer models (e.g., meteorological data) and the source of these data.

(8) A brief description of all construction and modifications which were completed in the calendar year for which the report is prepared, but for which the requirement to apply for approval to construct or modify was waived under § 61.96 and associated documentation developed by DOE to support the waiver. EPA reserves the right to require that DOE send to EPA all the information that normally would be required in an application to construct or modify, following receipt of the description and supporting documentation.

(9) Each report shall be signed and dated by a corporate officer or public official in charge of the facility and contain the following declaration immediately above the signature line: "I certify under penalty of law that I have personally examined and am familiar with the information submitted herein and based on my inquiry of those individuals immediately responsible for obtaining the information, I believe that the submitted information is true, accurate and complete. I am aware that there are significant penalties for submitting false information including the possibility of fine and imprisonment. See, 18 U.S.C. 1001."

(c) If the facility is not in compliance with the emission limits of § 61.92 in the calendar year covered by the report, then the facility must commence reporting to the Administrator on a monthly basis the information listed in paragraph (b) of this section, for the preceding month. These reports will start the month immediately following the submittal of the annual report for the year in noncompliance and will be due 30 days following the end of each month. This increased level of reporting will continue until the Administrator has determined that the monthly reports are no longer necessary. In addition to all the information required in paragraph (b) of this section, monthly reports shall also include the following information:

(1) All controls or other changes in operation of the facility that will be or

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are being installed to bring the facility into compliance.

(2) If the facility is under a judicial or administrative enforcement decree, the report will describe the facilities performance under the terms of the decree.

(d) In those instances where the information requested is classified, such information will be made available to EPA separate from the report and will be handled and controlled according to applicable security and classification regulations and requirements.

(Approved by the Office of Management and Budget under control number 2060-0191)

§61.95 Recordkeeping requirements.

All facilities must maintain records documenting the source of input parameters including the results of all measurements upon which they are based, the calculations and/or analytical methods used to derive values for input parameters, and the procedure used to determine effective dose equivalent. This documentation should be sufficient to allow an independent auditor to verify the accuracy of the determination made concerning the facility's compliance with the standard. These records must be kept at the site of the facility for at least five years and, upon request, be made available for inspection by the Administrator, or his authorized representative.

§ 61.96 Applications to construct or modify.

(a) In addition to any activity that is defined as construction under 40 CFR part 61, subpart A, any fabrication, erection or installation of a new building or structure within a facility that emits radionuclides is also defined as new construction for purposes of 40 CFR part 61, subpart A.

(b) An application for approval under § 61.07 or notification of startup under § 61.09 does not need to be filed for any new construction or modification within an existing facility if the effective dose equivalent, caused by all emissions from the new construction or modification, is less than 1% of the standard prescribed in § 61.92. For purposes of this paragraph the effective

dose equivalent shall be calculated using the source term derived using Appendix D as input to the dispersion and other computer models described in § 61.93. DOE may, with prior approval from EPA, use another procedure for estimating the source term for use in this paragraph. A facility is eligible for this exemption only if, based on its last annual report, the facility is in compliance with this subpart.

(c) Conditions to approvals granted under § 61.08 will not contain requirements for post approval reporting on operating conditions beyond those specified in § 61.94.

§ 61.97 Exemption from the reporting and testing requirements of 40 CFR 61.10.

All facilities designated under this subpart are exempt from the reporting requirements of 40 CFR 61.10.

Subpart I—National Emission Standards for Radionuclide Emissions From Facilities Licensed by the Nuclear Regulatory Commission and Federal Facilities Not Covered by Subpart H

SOURCE: 54 FR 51697, Dec. 15, 1989, unless otherwise noted.

EFFECTIVE DATE NOTE: At 54 FR 51697, Dec. 15, 1989, §§ 61.100 through 61.108 (subpart I) was revised and the effective date was stayed until March 15, 1990. At 55 FR 10455, Mar. 21, 1990, subpart I was further stayed until July 13, 1990. For the convenience of the user the superseded text appears after the new material below.

§ 61.100 Applicability.

The provisions of this subpart apply to Nuclear Regulatory Commission-licensed facilities and to facilities owned or operated by any Federal agency other than the Department of Energy, except that this subpart does not apply to disposal at facilities regulated under 40 CFR part 191, subpart B, or to any uranium mill tailings pile after it has been disposed of under 40 CFR part 192, or to low energy accelerators, or to any NRC-licensee that possesses and uses radionuclides only in the form of sealed sources.

§ 61.101 Definitions.

As used in this subpart, all terms not defined here have the meaning given them in the Clean Air Act or subpart A of part 61. The following terms shall have the following specific meanings:

(a) *Agreement State* means a State with which the Atomic Energy Commission or the Nuclear Regulatory Commission has entered into an effective agreement under subsection 274(b) of the Atomic Energy Act of 1954, as amended.

(b) *Effective dose equivalent* means the sum of the products of absorbed dose and appropriate factors to account for differences in biological effectiveness due to the quality of radiation and its distribution in the body of reference man. The unit of the effective dose equivalent is the rem. For purposes of this subpart doses caused by radon-222 and its decay products formed after the radon is released from the facility are not included. The method for calculating effective dose equivalent and the definition of reference man are outlined in the International Commission on Radiological Protection's Publication No. 26.

(c) *Facility* means all buildings, structures and operations on one contiguous site.

(d) *Federal facility* means any facility owned or operated by any department, commission, agency, office, bureau or other unit of the government of the United States of America except for facilities owned or operated by the Department of Energy.

(e) *NRC-licensed facility* means any facility licensed by the Nuclear Regulatory Commission or any Agreement State to receive title to, receive, possess, use, transfer, or deliver any source, by-product, or special nuclear material.

(f) *Radionuclide* means a type of atom which spontaneously undergoes radioactive decay.

§ 61.102 Standard.

(a) Emissions of radionuclides, including iodine, to the ambient air from a facility regulated under this subpart shall not exceed those amounts that would cause any member of the public to receive in any year an effective dose equivalent of 10 mrem/yr.

(b) Emissions of iodine to the ambient air from a facility regulated under this subpart shall not exceed those amounts that would cause any member of the public to receive in any year an effective dose equivalent of 3 mrem/yr.

§ 61.103 Determining compliance.

(a) Compliance with the emission standard in this subpart shall be determined through the use of either the EPA computer code COMPLY or the alternative requirements of Appendix E. Facilities emitting radionuclides not listed in COMPLY or Appendix E shall contact EPA to receive the information needed to determine dose. The source terms to be used for input into COMPLY shall be determined through the use of the measurement procedures listed in § 61.107 or the emission factors in Appendix D or through alternative procedures for which EPA has granted prior approval; or,

(b) Facilities may demonstrate compliance with the emission standard in this subpart through the use of computer models that are equivalent to COMPLY, provided that the model has received prior approval from EPA headquarters. Any facility using a model other than COMPLY must file an annual report. EPA may approve an alternative model in whole or in part and may limit its use to specific circumstances.

§ 61.104 Reporting requirements.

(a) The owner or operator of a facility subject to this subpart must submit an annual report to the EPA covering the emissions of a calendar year by March 31 of the following year.

(1) The report or application for approval to construct or modify as required by 40 CFR part 61, subpart A and § 61.106, must provide the following information:

(i) The name of the facility.

(ii) The name of the person responsible for the operation of the facility and the name of the person preparing the report (if different).

(iii) The location of the facility, including suite and/or building number, street, city, county, state, and zip code.

(iv) The mailing address of the facility, if different from item (iii).

(v) A list of the radioactive materials used at the facility.

(vi) A description of the handling and processing that the radioactive materials undergo at the facility.

(vii) A list of the stacks or vents or other points where radioactive materials are released to the atmosphere.

(viii) A description of the effluent controls that are used on each stack, vent, or other release point and an estimate of the efficiency of each device.

(ix) Distances from the point of release to the nearest residence, school, business or office and the nearest farms producing vegetables, milk, and meat.

(x) The effective dose equivalent calculated using the compliance procedures in § 61.103.

(xi) The physical form and quantity of each radionuclide emitted from each stack, vent or other release point, and the method(s) by which these quantities were determined.

(xii) The volumetric flow, diameter, effluent temperature, and release height for each stack, vent or other release point where radioactive materials are emitted, the method(s) by which these were determined.

(xiii) The height and width of each building from which radionuclides are emitted.

(xiv) The values used for all other user-supplied input parameters (e.g., meteorological data) and the source of these data.

(xv) A brief description of all construction and modifications which were completed in the calendar year for which the report is prepared, but for which the requirement to apply for approval to construct or modify was waived under section 61.106, and associated documentation developed by the licensee to support the waiver. EPA reserves the right to require that the licensee send to EPA all the information that normally would be required in an application to construct or modify, following receipt of the description and supporting documentation.

(xvi) Each report shall be signed and dated by a corporate officer or public official in charge of the facility and

contain the following declaration immediately above the signature line: "I certify under penalty of law that I have personally examined and am familiar with the information submitted herein and based on my inquiry of those individuals immediately responsible for obtaining the information, I believe that the submitted information is true, accurate and complete. I am aware that there are significant penalties for submitting false information including the possibility of fine and imprisonment. See, 18 U.S.C. 1001."

(b) Facilities emitting radionuclides in an amount that would cause less than 10% of the dose standard in § 61.102, as determined by the compliance procedures from § 61.103(a), are exempt from the reporting requirements of § 61.104(a). Facilities shall annually make a new determination whether they are exempt from reporting.

(c) If the facility is not in compliance with the emission limits of § 61.102 in the calendar year covered by the report, the facility must report to the Administrator on a monthly basis the information listed in paragraph (a) of this section, for the preceding month. These reports will start the month immediately following the submittal of the annual report for the year in noncompliance and will be due 30 days following the end of each month. This increased level of reporting will continue until the Administrator has determined that the monthly reports are no longer necessary. In addition to all the information required in paragraph (a) of this section, monthly reports shall also include the following information:

(1) All controls or other changes in operation of the facility that will be or are being installed to bring the facility into compliance.

(2) If the facility is under a judicial or administrative enforcement decree the report will describe the facilities performance under the terms of the decree.

(d) The first report will cover the emissions of calendar year 1990.

§ 61.105 Recordkeeping requirements.

The owner or operator of any facility must maintain records documenting the source of input parameters including the results of all measurements upon which they are based, the calculations and/or analytical methods used to derive values for input parameters, and the procedure used to determine compliance. This documentation should be sufficient to allow an independent auditor to verify the accuracy of the determination made concerning the facility's compliance with the standard, and, if claimed, qualification for exemption from reporting. These records must be kept at the site of the facility for at least five years and upon request be made available for inspection by the Administrator, or his authorized representative.

§ 61.106 Applications to construct or modify.

(a) In addition to any activity that is defined as construction under 40 CFR part 61, subpart A, any fabrication, erection or installation of a new building or structure within a facility is also defined as new construction for purposes of 40 CFR part 61, subpart A.

(b) An application under § 61.07 does not need to be filed for any new construction or modification within an existing facility if one of the following conditions is met:

(1) The effective dose equivalent calculated by using methods described in § 61.103, that is caused by all emissions from the facility including those potentially emitted by the proposed new construction or modification, is less than 10% of the standard prescribed in § 61.102.

(2) The effective dose equivalent calculated by using methods described in § 61.103, that is caused by all emissions from the new construction or modification, is less than 1% of the limit prescribed in § 61.102. A facility is eligible for this exemption only if the facility, based on its last annual report, is in compliance with this subpart.

§ 61.107 Emission determination.

(a) Facility owners or operators may, in lieu of monitoring, estimate radionuclide emissions in accordance with Appendix D, or other procedure for

which EPA has granted prior approval.

(b) Radionuclide emission rates from point sources (e.g. stacks or vents) shall be measured in accordance with the following requirements:

(1) Effluent flow rate measurements shall be made using the following methods:

(i) Reference Method 2 of Appendix A to part 60 shall be used to determine velocity and volumetric flow rates for stacks and large vents.

(ii) Reference Method 2A of Appendix A to part 60 shall be used to measure flow rates through pipes and small vents.

(iii) The frequency of the flow rate measurements shall depend upon the variability of the effluent flow rate. For variable flow rates, continuous or frequent flow rate measurements shall be made. For relatively constant flow rates only periodic measurements are necessary.

(2) Radionuclides shall be directly monitored or extracted, collected, and measured using the following methods:

(i) Reference Method 1 of Appendix A part 60 shall be used to select monitoring or sampling sites.

(ii) The effluent stream shall be directly monitored continuously using an in-line detector or representative samples of the effluent stream shall be withdrawn continuously from the sampling site following the guidance presented in ANSIN13.1-1969 "Guide to Sampling Airborne Radioactive Materials in Nuclear Facilities" (including the guidance presented in Appendix A of ANSIN13.1) (incorporated by reference—see § 61.18). The requirements for continuous sampling are applicable to batch processes when the unit is in operation. Periodic sampling (grab samples) may be used only with EPA's prior approval. Such approval may be granted in cases where continuous sampling is not practical and radionuclide emission rates are relatively constant. In such cases, grab samples shall be collected with sufficient frequency so as to provide a representative sample of the emissions.

(iii) Radionuclides shall be collected and measured using procedures based on the principles of measurement de-

scribed in Appendix B, Method 114. Use of methods based on principles of measurement different from those described in Appendix B, Method 114 must have prior approval from the Administrator. EPA reserves the right to approve alternative measurement procedures in whole or in part.

(iv) A quality assurance program shall be conducted that meets the performance requirements described in Appendix B, Method 114.

(3) When it is impractical to measure the effluent flow rate at an existing source in accordance with the requirements of paragraph (b)(1) of this section or to monitor or sample an effluent stream at an existing source in accordance with the site selection and sample extraction requirements of paragraph (b)(2) of this section, the facility owner or operator may use alternative effluent flow rate measurement procedures or site selection and sample extraction procedures provided that:

(i) It can be shown that the requirements of paragraphs (b) (1) and (2) of this section are impractical for the effluent stream.

(ii) The alternative procedure will not significantly underestimate the emissions.

(iii) The alternative procedure is fully documented.

(iv) The owner or operator has received prior approval from EPA.

(4)(i) Radionuclide emission measurements in conformance with the requirements of paragraph (b) of this section shall be made at all release points which have a potential to discharge radionuclides into the air in quantities which could cause an effective dose equivalent in excess of 1% of the standard. All radionuclides which could contribute greater than 10% of the potential effective dose equivalent for a release point shall be measured. For other release points which have a potential to release radionuclides into the air, periodic confirmatory measurements should be made to verify the low emissions.

(ii) To determine whether a release point is subject to the emission measurement requirements of paragraph (b) of this section, it is necessary to evaluate the potential for radionuclide

emissions for that release point. In evaluating the potential of a release point to discharge radionuclides into the air, the estimated radionuclide release rates shall be based on the discharge of the uncontrolled effluent stream into the air.

(5) Environmental measurements of radionuclide air concentrations at critical receptor locations may be used as an alternative to air dispersion calculations in demonstrating compliance with the standards if the owner or operator meets the following criteria:

(i) The air at the point of measurement shall be continuously sampled for collection of radionuclides.

(ii) Those radionuclides released from the facility, which are the major contributors to the effective dose equivalent must be collected and measured as part of the environmental measurements program.

(iii) Radionuclide concentrations which would cause an effective dose equivalent greater than or equal to 10% of the standard shall be readily detectable and distinguishable from background.

(iv) Net measured radionuclide concentrations shall be compared to the concentration levels in Table 2 of Appendix E to determine compliance with the standard. In the case of multiple radionuclides being released from a facility, compliance shall be demonstrated if the value for all radionuclides is less than the concentration level in Table 2 and the sum of the fractions that result when each measured concentration value is divided by the value in Table 2 for each radionuclide is less than 1.

(v) A quality assurance program shall be conducted that meets the performance requirements described in Appendix B, Method 114.

(vi) Use of environmental measurements to demonstrate compliance with the standard is subject to prior approval of EPA. Applications for approval shall include a detailed description of the sampling and analytical methodology and show how the above criteria will be met.

(c) The following facilities may use either the methodologies and quality assurance programs described in para-

graph (b) of this section or may use the following:

(1) Nuclear power reactors may determine their radionuclide emissions in conformance with the Effluent Technical Specifications contained in their Operating License issued by the Nuclear Regulatory Commission. In addition, they may conduct a quality assurance program as described in the Nuclear Regulatory Commission's Regulatory Guide 4.15 dated February 1979.

(2) Fuel processing and fabrication plants and uranium hexafluoride plants may determine their emissions in conformance with the Nuclear Regulatory Commission's Regulatory Guide 4.16 dated December 1985. In addition, they may conduct a quality assurance program as described in the Nuclear Regulatory Commission's Regulatory Guide 4.15 dated February 1979.

(3) Uranium mills may determine their emissions in conformance with the Nuclear Regulatory Commission's Regulatory Guide 4.14 dated April 1980. In addition, they may conduct a quality assurance program as described in the Nuclear Regulatory Commission's Regulatory Guide 4.15 dated February 1979.

§ 61.108 Exemption from the reporting and testing requirements of 40 CFR 61.10.

All facilities designated under this subpart are exempt from the reporting requirements of 40 CFR 61.10.

EFFECTIVE DATE NOTE: At 54 FR 51697, Dec. 15, 1989, §§ 61.100 through 61.108, subpart I was revised and the effective date was stayed until Mar. 15, 1990. At 55 FR 10455, Mar. 21, 1990, subpart I was further stayed until July 13, 1990. For the convenience of the user the superseded text appears below.

Subpart I—National Emission Standard for Radionuclide Emissions From Facilities Licensed by the Nuclear Regulatory Commission (NRC) and Federal Facilities Not Covered by Subpart H

SOURCE: 50 FR 5195, Feb. 5, 1985, unless otherwise noted.

EDITORIAL NOTE: The information collection requirements in §§ 61.107 and 61.108, added at 50 FR 5195, Feb. 5, 1985, have not been approved by the Office of Manage-

ment and Budget. Compliance with those provisions is not required until approved by OMB. EPA will publish a document in the **FEDERAL REGISTER** indicating the approval once it has been obtained.

§ 61.100 Designation of facilities.

The provisions of this subpart apply to NRC-licensed facilities and to facilities owned or operated by any Federal agency other than the Department of Energy that emit radionuclides to air. This subpart does not apply to facilities regulated under 40 CFR parts 190, 191, or 192, to any low energy accelerator, or to any user of the sealed radiation sources.

§ 61.101 Definitions.

(a) "Agreement State" means any State with which the Atomic Energy Commission or the Nuclear Regulatory Commission has entered into an effective agreement under subsection 274(b) of the Atomic Energy Act of 1954, as amended.

(b) "Dose equivalent" means the product of absorbed dose and appropriate factors to account for differences in biological effectiveness due to the quality of radiation and its distribution in the body. The unit of dose equivalent is the rem.

(c) "NRC-licensed facility" means any facility licensed by the Nuclear Regulatory Commission or any Agreement State to receive title to, receive, possess, use, transfer, or deliver any source, byproduct, or special nuclear material, except facilities regulated by 40 CFR parts 190, 191, or 192.

(d) "Critical organ" means the most exposed human organ or tissue exclusive of the integumentary system (skin) and the cornea.

(e) "Radionuclide" means any nuclide that emits radiation. (A nuclide is a species of atom characterized by the constitution of its nucleus and hence by the number of protons, the number of neutrons, and the energy content.)

(f) "Whole body" means all organs or tissues exclusive of the integumentary system (skin) and the cornea.

(g) "Effective dose equivalent" means the sum of the products of the dose equivalents to individual organs and tissues and appropriate weighting factors representing the risk relative to that for an equal dose to the whole body.

§ 61.102 Emission standard.

Emissions of radionuclides to air from facilities subject to this subpart shall not exceed those amounts that cause a dose equivalent of 25 mrem/y to the whole body or 75 mrem/y to the critical organ of any member of the public. Doses due to radon-220, radon-222, and their respective decay products are excluded from these limits.

§ 61.108

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§ 61.103 Emission monitoring and compliance procedures.

To determine compliance with the standard, radionuclide emissions shall be determined and dose equivalent to members of the public shall be calculated using EPA-approved sampling procedures, EPA codes AIRDOS-EPA and RADRISK, or other procedures, including those based on environmental measurements, that EPA has determined to be suitable. In most cases, compliance with this standard will be determined by calculating the dose to members of the public at the point of maximum annual air concentration in an unrestricted area where any member of the public resides or abides.

List of approved procedures: [Reserved]

§ 61.104 Reporting. [Reserved]

§ 61.105 Recordkeeping. [Reserved]

§ 61.106 Exemption from reporting and testing requirements of 40 CFR 61.10.

Facilities in possession of a radionuclide in annual quantities less than the activity shown in Table 1 are exempt from the reporting requirements of 40 CFR 61.10. If a facility possesses more than one radionuclide, and the sum of the annual amount possessed divided by the equivalent activity in Table 1 is summed for all radionuclides in possession, and the sum is less than unity, then the facility is exempt from the reporting requirements of 40 CFR 61.10. For radionuclides not on this list, a facility may apply to the Administrator for an exemption from the reporting requirements.

Table 1 [Reserved]

§ 61.107 Waiver of compliance.

(a) To request a waiver, applicants shall follow the requirements of § 61.10 (b)-(d).

(b) The following provisions also apply:

(1) the owner or operator of any existing source, or any new source to which a standard prescribed under this part is applicable which had an initial startup which preceded the effective date of a standard prescribed under this part shall, within 90 days after the effective date, provide the following information in writing to the Administrator:

(i) Name and address of the owner or operator.

(ii) The location of the source.

(iii) The types of radionuclides emitted by the stationary source and the annual quantity (in Ci/y for the most recent calendar year) of each radionuclide emitted.

(iv) A brief description of the nature, size, design, and method of operation of the stationary source including the operating design capacity of such source. Identify each point of emission for each hazardous pollutant.

(v) Estimate of dose equivalent rate to the member of the public at the point of maxi-

mum annual air concentration in an unrestricted area where any member of the public resides or abides.

(vi) A description of the existing control equipment for each emission point.

(A) Primary control device (s) for radionuclide emissions.

(B) Secondary control device(s) for radionuclide emissions.

(C) Estimated control efficiency (percent) for each control device.

(vii) A statement by the owner or operator of the source as to whether he can comply with the standards prescribed in this part within 90 days of the effective date.

§ 61.108 Alternative emission standard.

If a facility may exceed the emission standard established in § 61.102, the operator may apply to EPA for an alternative emission standard. The Administrator will review such applications and will establish an appropriate alternative emission standard that will ensure that no member of the public being exposed to emissions from the facility receives a continuous exposure of more than 100 mrem/y effective dose equivalent and a noncontinuous exposure of more than 500 mrem/y effective dose equivalent from all sources, excluding natural background and medical procedures. The application shall include the following:

(a) An assessment of the additional effective dose equivalents to the member of the public receiving maximum exposure from the facility due to all other sources. The natural radiation background shall be part of this assessment.

(b) The information required in § 61.107.

(c) The effective dose equivalent shall be calculated using the following weighting factors:

Organ	Weighting factor
[Reserved]	[Reserved]

Requests for alternative emission standards shall be sent to the Assistant Administrator for Air and Radiation (ANR-443), U.S. Environmental Protection Agency, 401 M Street SW., Washington, DC 20460. This action shall be taken, for existing facilities by April 17, 1985.

Subpart J—National Emission Standard for Equipment Leaks (Fugitive Emission Sources) of Benzene

SOURCE: 49 FR 23513, June 6, 1984, unless otherwise noted.

§ 61.110 Applicability and designation of sources.

(a) The provisions of this subpart apply to each of the following sources that are intended to operate in benzene service: pumps, compressors, pressure relief devices, sampling connections, systems, open-ended valves or lines, valves, flanges and other connectors, product accumulator vessels, and control devices or systems required by this subpart.

(b) The provisions of this subpart do not apply to sources located in coke by-product plants.

(c)(1) If an owner or operator applies for one of the exemptions in this paragraph, then the owner or operator shall maintain records as required in § 61.246(i).

(2) Any equipment in benzene service that is located at a plant site designed to produce or use less than 1,000 megagrams of benzene per year is exempt from the requirements of § 61.112.

(3) Any process unit (defined in § 61.241) that has no equipment in benzene service is exempt from the requirements of § 61.112.

(d) While the provisions of this subpart are effective, a source to which this subpart applies that is also subject to the provisions of 40 CFR part 60 only will be required to comply with the provisions of this subpart.

§ 61.111 Definitions.

As used in this subpart, all terms not defined herein shall have the meaning given them in the Act, in subpart A of part 61, or in subpart V of part 61, and the following terms shall have the specific meanings given them:

"In benzene service" means that a piece of equipment either contains or contacts a fluid (Liquid or gas) that is at least 10 percent benzene by weight as determined according to the provisions of § 61.245(d). The provisions of § 61.245(d) also specify how to determine that a piece of equipment is not in benzene service.

"Semiannual" means a 6-month period; the first semiannual period concludes on the last day of the last month during the 180 days following initial startup for new sources; and the first semiannual period concludes on

the last day of the last full month during the 180 days after June 6, 1984 for existing sources.

§ 61.112 Standards.

(a) Each owner or operator subject to the provisions of this subpart shall comply with the requirements of subpart V of this part.

(b) An owner or operator may elect to comply with the requirements of §§ 61.243-1 and 61.243-2.

(c) An owner or operator may apply to the Administrator for a determination of an alternative means of emission limitation that achieves a reduction in emissions of benzene at least equivalent to the reduction in emissions of benzene achieved by the controls required in this subpart. In doing so, the owner or operator shall comply with requirements of § 61.244.

Subpart K—National Emission Standards for Radionuclide Emissions From Elemental Phosphorus Plants

SOURCE: 54 FR 51699, Dec. 15, 1989, unless otherwise noted.

§ 61.120 Applicability.

The provisions of this subpart are applicable to owners or operators of calciners and nodulizing kilns at elemental phosphorus plants.

§ 61.121 Definitions.

(a) *Elemental phosphorus plant or plant* means any facility that processes phosphate rock to produce elemental phosphorus. A plant includes all buildings, structures, operations, calciners and nodulizing kilns on one contiguous site.

(b) *Calclner or Nodulizing kiln* means a unit in which phosphate rock is heated to high temperatures to remove organic material and/or to convert it to a nodular form. For the purpose of this subpart, calciners and nodulizing kilns are considered to be similar units.

§ 61.122 Emission standard.

Emissions of polonium-210 to the ambient air from all calciners and nodulizing kilns at an elemental phos-

phorus plant shall not exceed a total of 2 curies a year.

§ 61.123 Emission testing.

(a) Each owner or operator of an elemental phosphorus plant shall test emissions from the plant within 90 days of the effective date of this standard and annually thereafter. The Administrator may temporarily or permanently waive the annual testing requirement or increase the frequency of testing, if the Administrator determines that more testing is required.

(b) The Administrator shall be notified at least 30 days prior to an emission test so that EPA may, at its option, observe the test.

(c) An emission test shall be conducted at each operational calciner or nodulizing kiln. If emissions from a calciner or nodulizing kiln are discharged through more than one stack, then an emission test shall be conducted at each stack and the total emission rate from the calciner or kiln shall be the sum of the emission rates from each of the stacks.

(d) Each emission test shall consist of three sampling runs that meet the requirements of § 61.125. The phosphate rock processing rate during each run shall be recorded. An emission rate in curies per metric ton of phosphate rock processed shall be calculated for each run. The average of all three runs shall apply in computing the emission rate for the test. The annual polonium-210 emission rate from a calciner or nodulizing kiln shall be determined by multiplying the measured polonium-210 emission rate in curies per metric ton of phosphate rock processed by the annual phosphate rock processing rate in metric tons. In determining the annual phosphate rock processing rate, the values used for operating hours and operating capacity shall be values that will maximize the expected processing rate. For determining compliance with the emission standard of § 61.122, the total annual emission rate is the sum of the annual emission rates for all operating calciners and nodulizing kilns.

(e) If the owner or operator changes his operation in such a way as to increase his emissions of polonium-210, such as changing the type of rock

processed, the temperature of the calciners or kilns, or increasing the annual phosphate rock processing rate, then a new emission test, meeting the requirements of this section, shall be conducted within 45 days under these conditions.

(f) Each owner or operator of an elemental phosphorus plant shall furnish the Administrator with a written report of the results of the emission test within 60 days of conducting the test. The report must provide the following information:

(1) The name and location of the facility.

(2) The name of the person responsible for the operation of the facility and the name of the person preparing the report (if different).

(3) A description of the effluent controls that are used on each stack, vent, or other release point and an estimate of the efficiency of each device.

(4) The results of the testing, including the results of each sampling run completed.

(5) The values used in calculating the emissions and the source of these data.

(6) Each report shall be signed and dated by a corporate officer in charge of the facility and contain the following declaration immediately above the signature line: "I certify under penalty of law that I have personally examined and am familiar with the information submitted herein and based on my inquiry of those individuals immediately responsible for obtaining the information, I believe that the submitted information is true, accurate and complete. I am aware that there are significant penalties for submitting false information including the possibility of fine and imprisonment. See, 18 U.S.C. 1001."

(Approved by the Office of Management and Budget under control number 2060-0191)

§ 61.124 Recordkeeping requirements.

The owner or operator of any plant must maintain records documenting the source of input parameters including the results of all measurements upon which they are based, the calculations and/or analytical methods

used to derive values for input parameters, and the procedure used in emission testing. This documentation should be sufficient to allow an independent auditor to verify the accuracy of the results of the emission testing. These records must be kept at the site of the plant for at least five years and, upon request, be made available for inspection by the Administrator, or his authorized representative.

§ 61.125 Test methods and procedures.

(a) Each owner or operator of a source required to test emissions under § 61.123, unless an equivalent or alternate method has been approved by the Administrator, shall use the following test methods:

(1) Test Method 1 of Appendix A to 40 CFR part 60 shall be used to determine sample and velocity traverses;

(2) Test Method 2 of Appendix A to 40 CFR part 60 shall be used to determine velocity and volumetric flow rate;

(3) Test Method 3 of Appendix A to 40 CFR part 60 shall be used for gas analysis;

(4) Test Method 5 of Appendix A to 40 CFR part 60 shall be used to collect particulate matter containing the polonium-210; and

(5) Test Method 111 of Appendix B to 40 CFR part 61 shall be used to determine the polonium-210 emissions.

§ 61.126 Monitoring of operations.

(a) The owner or operator of any source subject to this subpart using a wet-scrubbing emission control device shall install, calibrate, maintain, and operate a monitoring device for the continuous measurement of the pressure loss of the gas stream through the scrubber. The monitoring device must be certified by the manufacturer to be accurate within ± 250 pascal (± 1 inch of water). Records of these measurements shall be maintained at the source and made available for inspection by the Administrator, or his authorized representative for a minimum of 5 years.

(b) The owner or operator of any source subject to this subpart using an electrostatic precipitator control device shall install, calibrate, maintain, and operate a monitoring device

for the continuous measurement of the primary and secondary current and the voltage in each electric field. Records of these measurements shall be maintained at the source and made available for inspection by the Administrator, or his authorized representative for a minimum of 5 years.

(c) For the purpose of conducting an emission test under § 61.123, the owner or operator of any source subject to the provisions of this subpart shall install, calibrate, maintain, and operate a device for measuring the phosphate rock feed to any affected calciner or nodulizing kiln. The measuring device used must be accurate to within ± 5 percent of the mass rate over its operating range. Records of these measurements shall be maintained at the source and made available for inspection by the Administrator, or his authorized representative for a minimum of 5 years.

§ 61.127 Exemption from the reporting and testing requirements of 40 CFR 61.10.

All facilities designated under this subpart are exempt from the reporting requirements of 40 CFR 61.10.

Subpart L—National Emission Standard for Benzene Emissions from Coke By-Product Recovery Plants

SOURCE: 54 FR 38073, Sept. 14, 1989, unless otherwise noted.

§ 61.130 Applicability and designation of sources.

(a) The provisions of this subpart apply to each of the following sources at furnace and foundry coke by-product recovery plants: tar decanters, tar storage tanks, tar-intercepting sumps, flushing-liquor circulation tanks, light-oil sumps, light-oil condensers, light-oil decanters, wash-oil decanters, wash-oil circulation tanks, naphthalene processing, final coolers, final-cooler cooling towers, and the following equipment that are intended to operate in benzene service: pumps, valves, exhausters, pressure relief devices, sampling connection systems, open-ended valves or lines, flanges or

other connectors, and control devices or systems required by § 61.135.

(b) The provisions of this subpart also apply to benzene storage tanks, BTX storage tanks, light-oil storage tanks, and excess ammonia-liquor storage tanks at furnace coke by-product recovery plants.

§ 61.131 Definitions.

As used in this subpart, all terms not defined herein shall have the meaning given them in the Act, in subpart A of part 61, and in subpart V of part 61. The following terms shall have the specific meanings given them:

Annual coke production means the coke produced in the batteries connected to the coke by-product recovery plant over a 12-month period. The first 12-month period concludes on the first December 31 that comes at least 12 months after the effective date or after the date of initial startup if initial startup is after the effective date.

Benzene storage tank means any tank, reservoir, or container used to collect or store refined benzene.

BTX storage tank means any tank, reservoir, or container used to collect or store benzene-toluene-xylene or other light-oil fractions.

Coke by-product recovery plant means any plant designed and operated for the separation and recovery of coal tar derivatives (by-products) evolved from coal during the coking process of a coke oven battery.

Equipment means each pump, valve, exhauster, pressure relief device, sampling connection system, open-ended valve or line, and flange or other connector in benzene service.

Excess ammonia-liquor storage tank means any tank, reservoir, or container used to collect or store a flushing liquor solution prior to ammonia or phenol recovery.

Exhauster means a fan located between the inlet gas flange and outlet gas flange of the coke oven gas line that provides motive power for coke oven gases.

Foundry coke means coke that is produced from raw materials with less than 26 percent volatile material by weight and that is subject to a coking period of 24 hours or more. Percent volatile material of the raw materials

(by weight) is the weighted average percent volatile material of all raw materials (by weight) charged to the coke oven per coking cycle.

Foundry coke by-product recovery plant means a coke by-product recovery plant connected to coke batteries whose annual coke production is at least 75 percent foundry coke.

Flushing-liquor circulation tank means any vessel that functions to store or contain flushing liquor that is separated from the tar in the tar decanter and is recirculated as the cooled liquor to the gas collection system.

Furnace coke means coke produced in by-product ovens that is not foundry coke.

Furnace coke by-product recovery plant means a coke by-product recovery plant that is not a foundry coke by-product recovery plant.

In benzene service means a piece of equipment, other than an exhauster, that either contains or contacts a fluid (liquid or gas) that is at least 10 percent benzene by weight or any exhauster that either contains or contacts a fluid (liquid or gas) at least 1 percent benzene by weight as determined by the provisions of § 61.137(b). The provisions of § 61.137(b) also specify how to determine that a piece of equipment is not in benzene service.

Light-oil condenser means any unit in the light-oil recovery operation that functions to condense benzene-containing vapors.

Light-oil decanter means any vessel, tank, or other type of device in the light-oil recovery operation that functions to separate light oil from water downstream of the light-oil condenser. A light-oil decanter also may be known as a light-oil separator.

Light-oil storage tank means any tank, reservoir, or container used to collect or store crude or refined light-oil.

Light-oil sump means any tank, pit, enclosure, or slop tank in light-oil recovery operations that functions as a wastewater separation device for hydrocarbon liquids on the surface of the water.

Naphthalene processing means any operations required to recover naphthalene including the separation, re-

fining, and drying of crude or refined naphthalene.

Process vessel means each tar decanter, flushing-liquor circulation tank, light-oil condenser, light-oil decanter, wash-oil decanter, or wash-oil circulation tank.

Semiannual means a 6-month period; the first semiannual period concludes on the last day of the last full month during the 180 days following initial startup for new sources; the first semiannual period concludes on the last day of the last full month during the 180 days after the effective date of the regulation for existing sources.

Tar decanter means any vessel, tank, or container that functions to separate heavy tar and sludge from flushing liquor by means of gravity, heat, or chemical emulsion breakers. A tar decanter also may be known as a flushing-liquor decanter.

Tar storage tank means any vessel, tank, reservoir, or other type of container used to collect or store crude tar or tar-entrained naphthalene, except for tar products obtained by distillation, such as coal tar pitch, creosotes, or carbolic oil. This definition also includes any vessel, tank, reservoir, or container used to reduce the water content of the tar by means of heat, residence time, chemical emulsion breakers, or centrifugal separation. A tar storage tank also may be known as a tar-dewatering tank.

Tar-intercepting sump means any tank, pit, or enclosure that serves to receive or separate tars and aqueous condensate discharged from the primary cooler. A tar-intercepting sump also may be known as a primary-cooler decanter.

Wash-oil circulation tank means any vessel that functions to hold the wash oil used in light-oil recovery operations or the wash oil used in the wash-oil final cooler.

Wash-oil decanter means any vessel that functions to separate, by gravity, the condensed water from the wash oil received from a wash-oil final cooler or from a light-oil scrubber.

§ 61.132 Standard: Process vessels, storage tanks, and tar-intercepting sumps.

(a)(1) Each owner or operator of a furnace or a foundry coke byproduct recovery plant shall enclose and seal all openings on each process vessel, tar storage tank, and tar intercepting sump.

(2) The owner or operator shall duct gases from each process vessel, tar storage tank, and tar-intercepting sump to the gas collection system, gas distribution system, or other enclosed point in the by-product recovery process where the benzene in the gas will be recovered or destroyed. This control system shall be designed and operated for no detectable emissions, as indicated by an instrument reading of less than 500 ppm above background and visual inspections, as determined by the methods specified in § 61.245(c). This system can be designed as a closed, positive pressure, gas blanketing system.

(i) Except, the owner or operator may elect to install, operate, and maintain a pressure relief device, vacuum relief device, an access hatch, and a sampling port on each process vessel, tar storage tank, and tar-intercepting sump. Each access hatch and sampling port must be equipped with a gasket and a cover, seal, or lid that must be kept in a closed position at all times, unless in actual use.

(ii) The owner or operator may elect to leave open to the atmosphere the portion of the liquid surface in each tar decanter necessary to permit operation of a sludge conveyor. If the owner or operator elects to maintain an opening on part of the liquid surface of the tar decanter, the owner or operator shall install, operate, and maintain a water leg seal on the tar decanter roof near the sludge discharge chute to ensure enclosure of the major portion of liquid surface not necessary for the operation of the sludge conveyor.

(b) Following the installation of any control equipment used to meet the requirements of paragraph (a) of this section, the owner or operator shall monitor the connections and seals on each control system to determine if it is operating with no detectable emis-

sions, using Reference Method 21 (40 CFR part 60, appendix A) and procedures specified in § 61.245(c), and shall visually inspect each source (including sealing materials) and the ductwork of the control system for evidence of visible defects such as gaps or tears. This monitoring and inspection shall be conducted on a semiannual basis and at any other time after the control system is repressurized with blanketing gas following removal of the cover or opening of the access hatch.

(1) If an instrument reading indicates an organic chemical concentration more than 500 ppm above a background concentration, as measured by Reference Method 21, a leak is detected.

(2) If visible defects such as gaps in sealing materials are observed during a visual inspection, a leak is detected.

(3) When a leak is detected, it shall be repaired as soon as practicable, but not later than 15 calendar days after it is detected.

(4) A first attempt at repair of any leak or visible defect shall be made no later than 5 calendar days after each leak is detected.

(c) Following the installation of any control system used to meet the requirements of paragraph (a) of this section, the owner or operator shall conduct a maintenance inspection of the control system on an annual basis for evidence of system abnormalities, such as blocked or plugged lines, sticking valves, plugged condensate traps, and other maintenance defects that could result in abnormal system operation. The owner or operator shall make a first attempt at repair within 5 days, with repair within 15 days of detection.

(d) Each owner or operator of a furnace coke by-product recovery plant also shall comply with the requirements of paragraphs (a)-(c) of this section for each benzene storage tank, BTX storage tank, light-oil storage tank, and excess ammonia-liquor storage tank.

§ 61.133 Standard: Light-oil sumps.

(a) Each owner or operator of a light-oil sump shall enclose and seal the liquid surface in the sump to form

a closed system to contain the emissions.

(1) Except, the owner or operator may elect to install, operate, and maintain a vent on the light-oil sump cover. Each vent pipe must be equipped with a water leg seal, a pressure relief device, or vacuum relief device.

(2) Except, the owner or operator may elect to install, operate, and maintain an access hatch on each light-oil sump cover. Each access hatch must be equipped with a gasket and a cover, seal, or lid that must be kept in a closed position at all times, unless in actual use.

(3) The light-oil sump cover may be removed for periodic maintenance but must be replaced (with seal) at completion of the maintenance operation.

(b) The venting of steam or other gases from the by-product process to the light-oil sump is not permitted.

(c) Following the installation of any control equipment used to meet the requirements of paragraph (a) of this section, the owner or operator shall monitor the connections and seals on each control system to determine if it is operating with no detectable emissions, using Reference Method 21 (40 CFR part 60, appendix A) and the procedures specified in § 61.245(c), and shall visually inspect each source (including sealing materials) for evidence of visible defects such as gaps or tears. This monitoring and inspection shall be conducted semiannually and at any other time the cover is removed.

(1) If an instrument reading indicates an organic chemical concentration more than 500 ppm above a background concentration, as measured by Reference Method 21, a leak is detected.

(2) If visible defects such as gaps in sealing materials are observed during a visual inspection, a leak is detected.

(3) When a leak is detected, it shall be repaired as soon as practicable, but not later than 15 calendar days after it is detected.

(4) A first attempt at repair of any leak or visible defect shall be made no later than 5 calendar days after each leak is detected.

§ 61.134 Standard: Naphthalene processing, final coolers, and final-cooler cooling towers.

(a) No ("zero") emissions are allowed from naphthalene processing, final coolers and final-cooler cooling towers at coke by-product recovery plants.

§ 61.135 Standard: Equipment leaks.

(a) Each owner or operator of equipment in benzene service shall comply with the requirements of 40 CFR 61, subpart V, except as provided in this section.

(b) The provisions of § 61.242-3 and § 61.242-9 of subpart V do not apply to this subpart.

(c) Each piece of equipment in benzene service to which this subpart applies shall be marked in such a manner that it can be distinguished readily from other pieces of equipment in benzene service.

(d) Each exhaustor shall be monitored quarterly to detect leaks by the methods specified in § 61.245(b) except as provided in § 61.136(d) and paragraphs (e)-(g) of this section.

(1) If an instrument reading of 10,000 ppm or greater is measured, a leak is detected.

(2) When a leak is detected, it shall be repaired as soon as practicable, but no later than 15 calendar days after it is detected, except as provided in § 61.242-10 (a) and (b). A first attempt at repair shall be made no later than 5 calendar days after each leak is detected.

(e) Each exhaustor equipped with a seal system that includes a barrier fluid system and that prevents leakage of process fluids to the atmosphere is exempt from the requirements of paragraph (d) of this section provided the following requirements are met:

(1) Each exhaustor seal system is:

(i) Operated with the barrier fluid at a pressure that is greater than the exhaustor stuffing box pressure; or

(ii) Equipped with a barrier fluid system that is connected by a closed vent system to a control device that complies with the requirements of § 61.242-11; or

(iii) Equipped with a system that purges the barrier fluid into a process stream with zero benzene emissions to the atmosphere.

(2) The barrier fluid is not in benzene service.

(3) Each barrier fluid system shall be equipped with a sensor that will detect failure of the seal system, barrier fluid system, or both.

(4)(i) Each sensor as described in paragraph (e)(3) of this section shall be checked daily or shall be equipped with an audible alarm.

(ii) The owner or operator shall determine, based on design considerations and operating experience, a criterion that indicates failure of the seal system, the barrier fluid system, or both.

(5) If the sensor indicates failure of the seal system, the barrier system, or both (based on the criterion determined under paragraph (e)(4)(ii) of this section), a leak is detected.

(6)(i) When a leak is detected, it shall be repaired as soon as practicable, but not later than 15 calendar days after it is detected, except as provided in § 61.242-10.

(ii) A first attempt at repair shall be made no later than 5 calendar days after each leak is detected.

(f) An exhaustor is exempt from the requirements of paragraph (d) of this section if it is equipped with a closed vent system capable of capturing and transporting any leakage from the seal or seals to a control device that complies with the requirements of § 61.242-11 except as provided in paragraph (g) of this section.

(g) Any exhaustor that is designated, as described in § 61.246(e) for no detectable emissions, as indicated by an instrument reading of less than 500 ppm above background, is exempt from the requirements of paragraph (d) of this section if the exhaustor:

(1) Is demonstrated to be operating with no detectable emissions, as indicated by an instrument reading of less than 500 ppm above background, as measured by the methods specified in § 61.245(c); and

(2) Is tested for compliance with paragraph (g)(1) of this section initially upon designation, annually, and at other times requested by the Administrator.

(h) Any exhaustor that is in vacuum service is excluded from the require-

§ 61.136

ments of this subpart if it is identified as required in § 61.246(e)(5).

§ 61.136 Compliance provisions and alternative means of emission limitation.

(a) Each owner or operator subject to the provisions of this subpart shall demonstrate compliance with the requirements of §§ 61.132 through 61.135 for each new and existing source, except as provided under §§ 61.243-1 and 61.243-2.

(b) Compliance with this subpart shall be determined by a review of records, review of performance test results, inspections, or any combination thereof, using the methods and procedures specified in § 61.137.

(c) On the first January 1 after the first year that a plant's annual coke production is less than 75 percent foundry coke, the coke by-product recovery plant becomes a furnace coke by-product recovery plant and shall comply with 61.132(d). Once a plant becomes a furnace coke by-product recovery plant, it will continue to be considered a furnace coke by-product recovery plant, regardless of the coke production in subsequent years.

(d)(1) An owner or operator may request permission to use an alternative means of emission limitation to meet the requirements in §§ 61.132, 61.133, and 61.135 of this subpart and §§ 61.242-2, -5, -6, -7, -8, and -11 of subpart V. Permission to use an alternative means of emission limitation shall be requested as specified in § 61.12(d).

(2) When the Administrator evaluates requests for permission to use alternative means of emission limitation for sources subject to §§ 61.132 and 61.133 (except tar decanters) the Administrator shall compare test data for the means of emission limitation to a benzene control efficiency of 98 percent. For tar decanters, the Administrator shall compare test data for the means of emission limitation to a benzene control efficiency of 95 percent.

(3) For any requests for permission to use an alternative to the work practices required under § 61.135, the provisions of § 61.244(c) shall apply.

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§ 61.137 Test methods and procedures.

(a) Each owner or operator subject to the provisions of this subpart shall comply with the requirements in § 61.245 of 40 CFR part 61, subpart V.

(b) To determine whether or not a piece of equipment is in benzene service, the methods in § 61.245(d) shall be used, except that, for exhausters, the percent benzene shall be 1 percent by weight, rather than the 10 percent by weight described in § 61.245(d).

§ 61.138 Recordkeeping and reporting requirements.

(a) The following information pertaining to the design of control equipment installed to comply with §§ 61.132 through 61.134 shall be recorded and kept in a readily accessible location:

(1) Detailed schematics, design specifications, and piping and instrumentation diagrams.

(2) The dates and descriptions of any changes in the design specifications.

(b) The following information pertaining to sources subject to § 61.132 and sources subject to § 61.133 shall be recorded and maintained for 2 years following each semiannual (and other) inspection and each annual maintenance inspection:

(1) The date of the inspection and the name of the inspector.

(2) A brief description of each visible defect in the source or control equipment and the method and date of repair of the defect.

(3) The presence of a leak, as measured using the method described in § 61.245(c). The record shall include the date of attempted and actual repair and method of repair of the leak.

(4) A brief description of any system abnormalities found during the annual maintenance inspection, the repairs made, the date of attempted repair, and the date of actual repair.

(c) Each owner or operator of a source subject to § 61.135 shall comply with § 61.246.

(d) For foundry coke by-product recovery plants, the annual coke production of both furnace and foundry coke

shall be recorded and maintained for 2 years following each determination.

(e)(1) An owner or operator of any source to which this subpart applies shall submit a statement in writing notifying the Administrator that the requirements of this subpart and 40 CFR 61, subpart V, have been implemented.

(2) In the case of an existing source or a new source that has an initial startup date preceding the effective date, the statement is to be submitted within 90 days of the effective date, unless a waiver of compliance is granted under § 61.11, along with the information required under § 61.10. If a waiver of compliance is granted, the statement is to be submitted on a date scheduled by the Administrator.

(3) In the case of a new source that did not have an initial startup date preceding the effective date, the statement shall be submitted with the application for approval of construction, as described under § 61.07.

(4) The statement is to contain the following information for each source:

(i) Type of source (e.g., a light-oil sump or pump).

(ii) For equipment in benzene service, equipment identification number and process unit identification; percent by weight benzene in the fluid at the equipment; and process fluid state in the equipment (gas/vapor or liquid).

(iii) Method of compliance with the standard (e.g., "gas blanketing," "monthly leak detection and repair," or "equipped with dual mechanical seals"). This includes whether the plant plans to be a furnace or foundry coke by-product recovery plant for the purposes of § 61.132(d).

(f) A report shall be submitted to the Administrator semiannually starting 6 months after the initial reports required in § 61.138(e) and § 61.10, which includes the following information:

(1) For sources subject to § 61.132 and sources subject to § 61.133,

(i) A brief description of any visible defect in the source or ductwork,

(ii) The number of leaks detected and repaired, and

(iii) A brief description of any system abnormalities found during

each annual maintenance inspection that occurred in the reporting period and the repairs made.

(2) For equipment in benzene service subject to § 61.135(a), information required by § 61.247(b).

(3) For each exhaustor subject to § 61.135 for each quarter during the semiannual reporting period,

(i) The number of exhaustors for which leaks were detected as described in § 61.135 (d) and (e)(5),

(ii) The number of exhaustors for which leaks were repaired as required in § 61.135 (d) and (e)(6),

(iii) The results of performance tests to determine compliance with § 61.135(g) conducted within the semiannual reporting period.

(4) A statement signed by the owner or operator stating whether all provisions of 40 CFR part 61, subpart L, have been fulfilled during the semiannual reporting period.

(5) For foundry coke by-product recovery plants, the annual coke production of both furnace and foundry coke, if determined during the reporting period.

(6) Revisions to items reported according to paragraph (e) of this section if changes have occurred since the initial report or subsequent revisions to the initial report.

NOTE: Compliance with the requirements of § 61.10(c) is not required for revisions documented under this paragraph.

(g) In the first report submitted as required in § 61.138(e), the report shall include a reporting schedule stating the months that semiannual reports shall be submitted. Subsequent reports shall be submitted according to that schedule unless a revised schedule has been submitted in a previous semiannual report.

(h) An owner or operator electing to comply with the provisions of §§ 61.243-1 and 61.243-2 shall notify the Administrator of the alternative standard selected 90 days before implementing either of the provisions.

(i) An application for approval of construction or modification, as required under §§ 61.05(a) and 61.07, will not be required for sources subject to 61.135 if:

(1) The new source complies with § 61.135, and

(2) In the next semiannual report required by § 61.138(f), the information described in § 61.138(e)(4) is reported.

(Approved by the Office of Management and Budget under control number 2060-0185)

[55 FR 38073, Sept. 14, 1990; 55 FR 14037, Apr. 13, 1990]

§ 61.139 Delegation of authority.

(a) In delegating implementation and enforcement authority to a State under Section 112(d) of the Act, the authorities contained in paragraph (b) of this section shall be retained by the Administrator and not transferred to a State.

(b) Authorities that will not be delegated to States: § 61.136(d).

Subpart M—National Emission Standard for Asbestos

SOURCE: 49 FR 13661, Apr. 5, 1984, unless otherwise noted.

§ 61.140 Applicability.

The provisions of this subpart are applicable to those sources specified in §§ 61.142 through 61.153.

§ 61.141 Definitions.

All terms that are used in this subpart and are not defined below are given the same meaning as in the Act and in subpart A of this part.

Active waste disposal site means any disposal site other than an inactive site.

Adequately wetted means sufficiently mixed or coated with water or an aqueous solution to prevent dust emissions.

Asbestos means the asbestiform varieties of serpentinite (chrysotile), riebeckite (crocidolite), cummingtonite-grunerite, anthophyllite, and actinolite-tremolite.

Asbestos-containing waste materials means any waste that contains commercial asbestos and is generated by a source subject to the provisions of this subpart. This term includes asbestos mill tailings, asbestos waste from control devices, friable asbestos waste material, and bags or containers that previously contained commercial as-

bestos. However, as applied to demolition and renovation operations, this term includes only friable asbestos waste and asbestos waste from control devices.

Asbestos material means asbestos or any material containing asbestos.

Asbestos mill means any facility engaged in converting, or in any intermediate step in converting, asbestos ore into commercial asbestos. Outside storage of asbestos material is not considered a part of the asbestos mill.

Asbestos tailings means any solid waste that contains asbestos and is a product of asbestos mining or milling operations.

Asbestos waste from control devices means any waste material that contains asbestos and is collected by a pollution control device.

Commercial asbestos means any asbestos that is extracted from asbestos ore.

Demolition means the wrecking or taking out of any load-supporting structural member of a facility together with any related handling operations.

Emergency renovation operation means a renovation operation that was not planned but results from a sudden, unexpected event. This term includes operations necessitated by nonroutine failures of equipment.

Fabricating means any processing of a manufactured product that contains commercial asbestos, with the exception of processing at temporary sites for the construction or restoration of facilities.

Facility means any institutional, commercial, or industrial structure, installation, or building (excluding apartment buildings having no more than four dwelling units).

Facility component means any pipe, duct, boiler, tank, reactor, turbine, or furnace at or in a facility; or any structural member of a facility.

Friable asbestos material means any material containing more than 1 percent asbestos by weight that hand pressure can crumble, pulverize, or reduce to powder when dry.

Inactive waste disposal site means any disposal site or portion of it where additional asbestos-containing waste material will not be deposited and

where the surface is not disturbed by vehicular traffic.

Manufacturing means the combining of commercial asbestos—or, in the case of woven friction products, the combining of textiles containing commercial asbestos—with any other material(s), including commercial asbestos, and the processing of this combination into a product.

Outside air means the air outside buildings and structures.

Particulate asbestos material means finely divided particles of asbestos material.

Planned renovation operations means a renovation operation, or a number of such operations, in which the amount of friable asbestos material that will be removed or stripped within a given period of time can be predicted. Individual nonscheduled operations are included if a number of such operations can be predicted to occur during a given period of time based on operating experience.

Remove means to take out friable asbestos materials from any facility.

Renovation means altering in any way one or more facility components. Operations in which load-supporting structural members are wrecked or taken out are excluded.

Roadways means surfaces on which motor vehicles travel. This term includes highways, roads, streets, parking areas, and driveways.

Strip means to take off friable asbestos materials from any part of a facility.

Structural member means any load-supporting member of a facility, such as beams and load supporting walls; or any nonload-supporting member, such as ceilings and nonload-supporting walls.

Visible emissions means any emissions containing particulate asbestos material that are visually detectable without the aid of instruments. This does not include condensed uncombined water vapor.

[49 FR 13661, Apr. 5, 1984; 49 FR 25453, June 21, 1984]

§ 61.142 Standard for asbestos mills.

Each owner or operator of an asbestos mill shall either discharge no visible emissions to the outside air from

that asbestos mill or use the methods specified by § 61.154 to clean emissions containing particulate asbestos material before they escape to, or are vented to, the outside air.

§ 61.143 Standard for roadways.

No person may surface a roadway with asbestos tailings or asbestos-containing waste material on that roadway, unless it is a temporary roadway on an area of asbestos ore deposits.

[49 FR 13661, Apr. 5, 1984; 49 FR 25453, June 21, 1984]

§ 61.144 Standard for manufacturing.

(a) **Applicability.** This section applies to the following manufacturing operations using commercial asbestos.

(1) The manufacture of cloth, cord, wicks, tubing, tape, twine, rope, thread, yarn, roving, lap, or other textile materials.

(2) The manufacture of cement products.

(3) The manufacture of fireproofing and insulating materials.

(4) The manufacture of friction products.

(5) The manufacture of paper, millboard, and felt.

(6) The manufacture of floor tile.¹

(7) The manufacture of paints, coatings, caulks, adhesives, and sealants.

(8) The manufacture of plastics and rubber materials.

(9) The manufacture of chlorine.

(10) The manufacture of shotgun shell wads.

(11) The manufacture of asphalt concrete.

(b) **Standard.** Each owner or operator of any of the manufacturing operations to which this section applies shall either:

(1) Discharge no visible emissions to the outside air from these operations or from any building or structure in which they are conducted; or

(2) Use the methods specified by § 61.154 to clean emissions from these operations containing particulate asbestos material before they escape to, or are vented to, the outside air.

§ 61.145 Standard for demolition and renovation: Applicability.

The requirements of §§ 61.146 and 61.147 apply to each owner or operator of a demolition or renovation operation as follows:

(a) If the amount of friable asbestos materials in a facility being demolished is at least 80 linear meters (260 linear feet) on pipes or at least 15 square meters (160 square feet) on other facility components, all the requirements of §§ 61.146 and 61.147 apply, except as provided in paragraph (c) of this section.

(b) If the amount of friable asbestos materials in a facility being demolished is less than 80 linear meters (260 linear feet) on pipes and less than 15 square meters (160 square feet) on other facility components, only the requirements of paragraphs (a), (b), and (c) (1), (2), (3), (4), and (5) of § 61.146 apply.

(c) If the facility is being demolished under an order of a State or local governmental agency, issued because the facility is structurally unsound and in danger of imminent collapse, only the requirements in § 61.146 and in paragraphs (d), (e), (f), and (g) of § 61.147 apply.

(d) If at least 80 linear meters (260 linear feet) of friable asbestos materials on pipes or at least 15 square meters (160 square feet) of friable asbestos materials on other facility components are stripped or removed at a facility being renovated, all the requirements of §§ 61.146 and 61.147 apply.

(1) To determine whether paragraph (d) of this section applies to planned renovation operations involving individual nonscheduled operations, predict the additive amount of friable asbestos materials to be removed or stripped over the maximum period of time a prediction can be made, not to exceed 1 year.

(2) To determine whether paragraph (d) of this section applies to emergency renovation operations, estimate the amount of friable asbestos materials to be removed or stripped as a result of the sudden, unexpected event that necessitated the renovation.

(e) Owners or operators of demolition and renovation operations are

exempt from the requirements of §§ 61.05(a), 61.07, and 61.09.

[49 FR 13661, Apr. 5, 1984; 49 FR 25453, June 21, 1984]

§ 61.146 Standard for demolition and renovation: Notification requirements.

Each owner or operator to which this section applies shall:

(a) Provide the Administrator with written notice of intention to demolish or renovate.

(b) Postmark or deliver the notice as follows:

(1) At least 10 days before demolition begins if the operation is described in § 61.145(a);

(2) At least 20 days before demolition begins if the operation is described in § 61.145(b);

(3) As early as possible before demolition begins if the operation is described in § 61.145(c);

(4) As early as possible before renovation begins.

(c) Include the following information in the notice:

(1) Name and address of owner or operator.

(2) Description of the facility being demolished or renovated, including the size, age, and prior use of the facility.

(3) Estimate of the approximate amount of friable asbestos material present in the facility in terms of linear feet of pipe, and surface area on other facility components. For facilities described in § 61.145(b), explain techniques of estimation.

(4) Location of the facility being demolished or renovated.

(5) Scheduled starting and completion dates of demolition or renovation.

(6) Nature of planned demolition or renovation and method(s) to be used.

(7) Procedures to be used to comply with the requirements of this subpart.

(8) Name and location of the waste disposal site where the friable asbestos waste material will be deposited.

(9) For facilities described in § 61.145(c), the name, title, and authority

of the State or local governmental representative who has ordered the demolition.

(Approved by the Office of Management and Budget under control number 2000-0264)

[49 FR 13661, Apr. 5, 1984; 49 FR 25453, June 21, 1984]

§ 61.147 Standard for demolition and renovation: Procedures for asbestos emission control.

Each owner or operator to whom this section applies shall comply with the following procedures to prevent emissions of particulate asbestos material to the outside air:

(a) Remove friable asbestos materials from a facility being demolished or renovated before any wrecking or dismantling that would break up the materials or preclude access to the materials for subsequent removal. However, friable asbestos materials need not be removed before demolition if:

(1) They are on a facility component that is encased in concrete or other similar material; and

(2) These materials are adequately wetted whenever exposed during demolition.

(b) When a facility component covered or coated with friable asbestos materials is being taken out of the facility as units or in sections:

(1) Adequately wet any friable asbestos materials exposed during cutting or disjoining operations; and

(2) Carefully lower the units or sections to ground level, not dropping them or throwing them.

(c) Adequately wet friable asbestos materials when they are being stripped from facility components before the members are removed from the facility. In renovation operations, wetting that would unavoidably damage equipment is not required if the owner or operator:

(1) Asks the Administrator to determine whether wetting to comply with this paragraph would unavoidably damage equipment, and, before beginning to strip, supplies the Administrator with adequate information to make this determination; and

(2) When the Administrator does determine that equipment damage would be unavoidable, uses a local exhaust ventilation and collection system designed and operated to capture the particulate asbestos material produced by the stripping and removal of the

friable asbestos materials. The system must exhibit no visible emissions to the outside air or be designed and operated in accordance with the requirements in § 61.154.

(d) After a facility component has been taken out of the facility as units or in sections, either:

(1) Adequately wet friable asbestos materials during stripping; or

(2) Use a local exhaust ventilation and collection system designed and operated to capture the particulate asbestos material produced by the stripping. The system must exhibit no visible emissions to the outside air or be designed and operated in accordance with the requirements in § 61.154.

(e) For friable asbestos materials that have been removed or stripped:

(1) Adequately wet the materials to ensure that they remain wet until they are collected for disposal in accordance with § 61.152; and

(2) Carefully lower the materials to the ground or a lower floor, not dropping or throwing them; and

(3) Transport the materials to the ground via dust-tight chutes or containers if they have been removed or stripped more than 50 feet above ground level and were not removed as units or in sections.

(f) When the temperature at the point of wetting is below 0°C (32°F):

(1) Comply with the requirements of paragraphs (d) and (e) of this section. The owner or operator need not comply with the other wetting requirements in this section; and

(2) Remove facility components coated or covered with friable asbestos materials as units or in sections to the maximum extent possible.

(g) For facilities described in § 61.145(c), adequately wet the portion of the facility that contains friable asbestos materials during the wrecking operation.

§ 61.148 Standard for spraying.

The owner or operator of an operation in which asbestos-containing materials are spray applied shall comply with the following requirements:

(a) Use materials that contain 1 percent asbestos or less on a dry weight basis for spray-on application on build-

ings, structures, pipes, and conduits, except as provided in paragraph (c) of this section.

(b) For spray-on application of materials that contain more than 1 percent asbestos on a dry weight basis on equipment and machinery, except as provided in paragraph (c) of this section:

(1) Notify the Administrator at least 20 days before beginning the spraying operation. Include the following information in the notice:

(i) Name and address of owner or operator.

(ii) Location of spraying operation.

(iii) Procedures to be followed to meet the requirements of this paragraph.

(2) Discharge no visible emissions to the outside air from the spray-on application of the asbestos-containing material or use the methods specified by § 61.154 to clean emissions containing particulate asbestos material before they escape to, or are vented to, the outside air.

(c) The requirements of paragraphs (a) and (b) of this section do not apply to the spray-on application of materials where the asbestos fibers in the materials are encapsulated with a bituminous or resinous binder during spraying and the materials are not friable after drying.

(d) Owners and operators of sources subject to this section are exempt from the requirements of §§ 61.05(a), 61.07, and 61.09.

(Approved by the Office of Management and Budget under control number 2000-0264)

§ 61.149 Standard for fabricating.

(a) *Applicability.* This section applies to the following fabricating operations using commercial asbestos:

(1) The fabrication of cement building products.

(2) The fabrication of friction products, except those operations that primarily install asbestos friction materials on motor vehicles.

(3) The fabrication of cement or silicate board for ventilation hoods; ovens; electrical panels; laboratory furniture, bulkheads, partitions; and ceilings for marine construction; and

flow control devices for the molten metal industry.

(b) *Standard.* Each owner or operator of any of the fabricating operations to which this section applies shall either:

(1) Discharge no visible emissions to the outside air from any of the operations or from any building or structure in which they are conducted; or

(2) Use the methods specified by § 61.154 to clean emissions containing particulate asbestos material before they escape to, or are vented to, the outside air.

§ 61.150 Standard for insulating materials.

After the effective date of this regulation, no owner or operator of a facility may install or reinstall on a facility component any insulating materials that contain commercial asbestos if the materials are either molded and friable or wet-applied and friable after drying. The provisions of this paragraph do not apply to spray-applied insulating materials regulated under § 61.148.

§ 61.151 Standard for waste disposal for asbestos mills.

Each owner or operator of any source covered under the provisions of § 61.142 shall:

(a) Deposit all asbestos-containing waste material at waste disposal sites operated in accordance with the provisions of § 61.156; and

(b) Discharge no visible emissions to the outside air from the transfer of asbestos waste from control devices to the tailings conveyor, or use the methods specified by § 61.154 to clean emissions containing particulate asbestos material before they escape to, or are vented to, the outside air. Dispose of the asbestos waste from control devices in accordance with § 61.152(b) or paragraph (c) of this section; and

(c) Discharge no visible emissions to the outside air during the collection, processing, packaging, transporting, or deposition of any asbestos-containing waste material, or use one of the disposal methods specified in paragraphs (c) (1) or (2) of this section, as follows:

(1) Use a wetting agent as follows:

(i) Adequately mix all asbestos-containing waste material with a wetting agent recommended by the manufacturer of the agent to effectively wet dust and tailings, before depositing the material at a waste disposal site. Use the agent as recommended for the particular dust by the manufacturer of the agent.

(ii) Discharge no visible emissions to the outside air from the wetting operation or use the methods specified by § 61.154 to clean emissions containing particulate asbestos material before they escape to, or are vented to, the outside air.

(iii) Wetting may be suspended when the ambient temperature at the waste disposal site is less than -9.5°C (15°F). Determine the ambient air temperature by an appropriate measurement method with an accuracy of $\pm^{\circ}\text{C}$ ($\pm 2^{\circ}\text{F}$), and record it at least hourly while the wetting operation is suspended. Keep the records for at least 2 years in a form suitable for inspection.

(2) Use an alternative disposal method that has received prior approval by the Administrator.

§ 61.152 Standard for waste disposal for manufacturing demolition, renovation, spraying, and fabricating operations.

Each owner or operator of any source covered under the provisions of § 61.144, 61.147, 61.148 and 61.149 shall:

(a) Deposit all asbestos-containing waste material at waste disposal sites operated in accordance with the provisions of § 61.156; and

(b) Discharge no visible emissions to the outside air during the collection, processing (including incineration), packaging, transporting, or deposition of any asbestos-containing waste material generated by the source, or use one of the disposal methods specified in paragraphs (b)(1), (2), or (3) of this section, as follows:

(1) Treat asbestos-containing waste material with water:

(i) Mix asbestos waste from control devices with water to form a slurry; adequately wet other asbestos-containing waste material; and

(ii) Discharge no visible emissions to the outside air from collection, mixing,

and wetting operations, or use the methods specified by § 61.154 to clean emissions containing particulate asbestos material before they escape to, or are vented to, the outside air; and

(iii) After wetting, seal all asbestos-containing waste material in leak-tight containers while wet; and

(iv) Label the containers specified in paragraph (b)(1)(iii) as follows:

CAUTION

Contains Asbestos-
Avoid Opening or
Breaking Container
Breathing Asbestos is Hazardous
to Your Health

Alternatively, use warning labels specified by Occupational Safety and Health Standards of the Department of Labor, Occupational Safety and Health Administration (OSHA) under 29 CFR 1910.1001(g)(2)(ii).

(2) Process asbestos-containing waste material into nonfriable forms:

(i) Form all asbestos-containing waste material into nonfriable pellets or other shapes; and

(ii) Discharge no visible emissions to the outside air from collection and processing operations, or use the methods specified by § 61.154 to clean emissions containing particulate asbestos material before they escape to, or are vented to, the outside air.

(3) Use an alternative disposal method that has received prior approval by the Administrator.

[49 FR 13661, Apr. 5, 1984; 49 FR 25453, June 21, 1984, as amended at 51 FR 8199, Mar. 10, 1986]

§ 61.153 Standard for inactive waste disposal sites for asbestos mills and manufacturing and fabricating operations.

Each owner or operator of any inactive waste disposal site that was operated by sources covered under § 61.142, § 61.144, or § 61.149 and received deposits of asbestos-containing waste material generated by the sources, shall

(a) Comply with one of the following:

(1) Either discharge no visible emissions to the outside air from an inac-

§ 61.154

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tive waste disposal site subject to this paragraph; or

(2) Cover the asbestos-containing waste material with at least 15 centimeters (6 inches) of compacted nonasbestos-containing material, and grow and maintain a cover of vegetation on the area adequate to prevent exposure of the asbestos-containing waste material; or

(3) Cover the asbestos-containing waste material with at least 60 centimeters (2 feet) of compacted nonasbestos-containing material, and maintain it to prevent exposure of the asbestos-containing waste; or

(4) For inactive waste disposal sites for asbestos tailings, apply a resinous or petroleum-based dust suppression agent that effectively binds dust and controls wind erosion. Use the agent as recommended for the particular asbestos tailings by the manufacturer of the dust suppression agent. Obtain prior approval of the Administrator to use other equally effective dust suppression agents. For purposes of this paragraph, waste crankcase oil is not considered a dust suppression agent.

(b) Unless a natural barrier adequately deters access by the general public, install and maintain warning signs and fencing as follows, or comply with paragraph (a)(2) or (a)(3) of this section.

(1) Display warning signs at all entrances and at intervals of 100 m (328 ft) or less along the property line of the site or along the perimeter of the sections of the site where asbestos-containing waste material was deposited. The warning signs must:

(i) Be posted in such a manner and location that a person can easily read the legend; and

(ii) Conform to the requirements for 51 cm×36 cm (20"×14") upright format signs specified in 29 CFR 1910.145(d)(4) and this paragraph; and

(iii) Display the following legend in the lower panel with letter sizes and styles of a visibility at least equal to those specified in this paragraph.

Legend	Notation
Asbestos Waste Disposal Sta.	2.5 cm (1 inch) Sans Serif, Gothic or Block

Legend	Notation
Do Not Create Dust	1.9 cm (¾ inch) Sans Serif, Gothic or Block
Breathing Asbestos is Hazardous to Your Health	14 Point Gothic.

Spacing between any two lines must be at least equal to the height of the upper of the two lines.

(2) Fence the perimeter of the site in a manner adequate to deter access by the general public.

(3) Upon request and supply of appropriate information, the Administrator will determine whether a fence or a natural barrier adequately deters access by the general public.

(c) The owner or operator may use an alternative control method that has received prior approval of the Administrator rather than comply with the requirements of paragraph (a) or (b) of this section.

[49 FR 13661, Apr. 5, 1984, as amended at 53 FR 36972, Sept. 23, 1988]

§ 61.154 Air-cleaning.

(a) The owner or operator who elects to use air-cleaning, as permitted by §§ 61.142, 61.144(b)(2), 61.147(c)(2), 61.147(d)(2), 61.148(b)(2), 61.149(b)(2), 61.151(b), 61.151(c)(1)(ii), 61.152(b)(1)(ii), and 61.152(b)(2) shall:

(1) Use fabric filter collection devices, except as noted in paragraph (b) of this section, doing all of the following:

(i) Operating the fabric filter collection devices at a pressure drop of no more than .995 kilopascal (4 inches water gage), as measured across the filter fabric; and

(ii) Ensuring that the airflow permeability, as determined by ASTM Method D737-75, does not exceed 9 m³/min/m² (30 ft³/min/ft²) for woven fabrics or 11³/min/m² (35 ft³/min/ft²) for felted fabrics, except that 12 m³/min/m² (40 ft³/min/ft²) for woven and 14 m³/min/m² (45 ft³/min/ft²) for felted fabrics is allowed for filtering air from asbestos ore dryers; and

(iii) Ensuring that felted fabric weighs at least 475 grams per square meter (14 ounces per square yard) and is at least 1.6 millimeters (one-sixteenth inch) thick throughout; and

(iv) Avoiding the use of synthetic fabrics that contain fill yarn other than that which is spun.

(2) Properly install, use, operate, and maintain all air-cleaning equipment authorized by this section. Bypass devices may be used only during upset or emergency conditions and then only for so long as it takes to shut down the operation generating the particulate asbestos material.

(b) There are the following exceptions to paragraph (a)(1):

(1) If the use of fabric creates a fire or explosion hazard, the Administrator may authorize as a substitute the use of wet collectors designed to operate with a unit contacting energy of at least 9.95 kilopascals (40 inches water gage pressure).

(2) The Administrator may authorize the use of filtering equipment other than that described in paragraphs (a)(1) and (b)(1) of this section if the owner or operator demonstrates to the Administrator's satisfaction that it is equivalent to the described equipment in filtering particulate asbestos material.

[49 FR 13661, Apr. 5, 1984; 49 FR 25453, June 21, 1984, as amended at 51 FR 8199, Mar. 10, 1986]

§ 61.155 Reporting.

(a) Within 90 days after the effective date of this subpart, each owner or operator of any existing source to which this subpart applies shall provide the following information to the Administrator, except that any owner or operator who provided this information prior to April 5, 1984 in order to comply with § 61.24 (which this section replaces) is not required to resubmit it.

(1) A description of the emission control equipment used for each process; and

(2) If a fabric filter device is used to control emissions, the pressure drop across the fabric filter in inches water gage; and

(i) If the fabric device uses a woven fabric, the airflow permeability in $\text{m}^3/\text{min}/\text{m}^2$ and; if the fabric is synthetic, whether the fill yarn is spun or not spun; and

(ii) If the fabric filter device uses a felted fabric, the density in g/m^2 , the

minimum thickness in inches, and the airflow permeability in $\text{m}^3/\text{min}/\text{m}^2$.

(3) For sources subject to §§ 61.151 and 61.152:

(i) A brief description of each process that generates asbestos-containing waste material; and

(ii) The average weight of asbestos-containing waste material disposed of, measured in kg/day ; and

(iii) The emission control methods used in all stages of water disposal; and

(iv) The type of disposal site or incineration site used for ultimate disposal, the name of the site operator, and the name and location of the disposal site.

(4) For sources subject to § 61.153:

(i) A brief description of the site; and

(ii) The method or methods used to comply with the standard, or alternative procedures to be used.

(b) The information required by paragraph (a) of this section must accompany the information required by § 61.10. The information described in this section must be reported using the format of Appendix A of this part.

(Approved by the Office of Management and Budget under control number 2000-0264)

(Sec. 114, Clean Air Act as amended (42 U.S.C. 7414))

§ 61.156 Active waste disposal sites.

To be an acceptable site for disposal of asbestos-containing waste material under §§ 61.151 and 61.152, an active waste disposal site must meet the requirements of this section.

(a) Either there must be no visible emissions to the outside air from any active waste disposal site where asbestos-containing waste material has been deposited, or the requirements of paragraph (c) or (d) of this section must be met.

(b) Unless a natural barrier adequately deters access by the general public, either warning signs and fencing must be installed and maintained as follows, or the requirements of paragraph (c)(1) of this section must be met.

(1) Warning signs must be displayed at all entrances and at intervals of 100

m (330 ft) or less along the property line of the site or along the perimeter of the sections of the site where asbestos-containing waste material is deposited. The warning signs must:

(i) Be posted in such a manner and location that a person can easily read the legend; and

(ii) Conform to the requirements of 51 cm x 36 cm (20" x 14") upright format signs specified in 29 CFR 1910.145(d)(4) and this paragraph; and

(iii) Display the following legend in the lower panel with letter sizes and styles of a visibility at least equal to those specified in this paragraph.

Legend	Notation
Asbestos Waste Disposal Site.	2.5 cm (1 inch) Sans Serif, Gothic or Block.
Do Not Create Dust.....	1.9 cm (¾ inch) Sans Serif, Gothic or Block.
Breathing Asbestos is Hazardous to Your Health.	14 Point Gothic.

Spacing between any two lines must be at least equal to the height of the upper of the two lines.

(2) The perimeter of the disposal site must be fenced in a manner adequate to deter access by the general public.

(3) Upon request and supply of appropriate information, the Administrator will determine whether a fence or a natural barrier adequately deters access by the general public.

(c) Rather than meet the no visible emission requirement of paragraph (a) of this section, an active waste disposal site would be an acceptable site if at the end of each operating day, or at least once every 24-hour period while the site is in continuous operation, the asbestos-containing waste material which was deposited at the site during the operating day or previous 24-hour period is covered with either.

(1) At least 15 centimeters (6 inches) of compacted nonasbestos-containing material, or

(2) A resinous or petroleum-based dust suppression agent that effectively binds dust and controls wind erosion. This agent must be used as recommended for the particular dust by the manufacturer of the dust suppression agent. Other equally effective dust suppression agents may be used upon

prior approval by the Administrator. For purposes of this paragraph, waste crankcase oil is not considered a dust suppression agent.

(d) Rather than meet the no visible emission requirement of paragraph (a) of this section, an active waste disposal site would be an acceptable site if an alternative control method for emissions that has received prior approval by the Administrator is used.

(Secs. 112 and 301(a) of the Clean Air Act as amended (42 U.S.C. 7412, 7601(a))

Subpart N—National Emission Standard for Inorganic Arsenic Emissions from Glass Manufacturing Plants

SOURCE: 51 FR 28025, Aug. 4, 1986, unless otherwise noted.

§ 61.160 Applicability and designation of source.

(a) The source to which this subpart applies is each glass melting furnace that uses commercial arsenic as a raw material. This subpart does not apply to pot furnaces.

(b) Rebrickng is not considered construction or modification for the purposes of § 61.05(a).

§ 61.161 Definitions.

The terms used in this subpart are defined in the Clean Air Act, in § 61.02, or in this section as follows:

"Arsenic-containing glass type" means any glass that is distinguished from other glass solely by the weight percent of arsenic added as a raw material and by the weight percent of arsenic in the glass produced. Any two or more glasses that have the same weight percent of arsenic in the raw materials as well as in the glass produced shall be considered to belong to one arsenic-containing glass type, without regard to the recipe used or any other characteristics of the glass or the method of production.

"By-pass the control device" means to operate the glass melting furnace without operating the control device to which that furnace's emissions are directed routinely.

"Commercial arsenic" means any form of arsenic that is produced by ex-

traction from any arsenic-containing substance and is intended for sale or for intentional use in a manufacturing process. Arsenic that is a naturally occurring trace constituent of another substance is not considered "commercial arsenic."

"Cullet" means waste glass recycled to a glass melting furnace.

"Glass melting furnace" means a unit comprising a refractory vessel in which raw materials are charged, melted at high temperature, refined, and conditioned to produce molten glass. The unit includes foundations, superstructure and retaining walls, raw material charger systems, heat exchangers, melter cooling system, exhaust system, refractory brick work, fuel supply and electrical boosting equipment, integral control systems and instrumentation, and appendages for conditioning and distributing molten glass to forming apparatuses. The forming apparatuses, including the float bath used in flat glass manufacturing, are not considered part of the glass melting furnace.

"Glass produced" means the glass pulled from the glass melting furnace.

"Inorganic arsenic" means the oxides and other noncarbon compounds of the element arsenic included in particulate matter, vapors, and aerosols.

"Malfunction" means any sudden failure of air pollution control equipment or process equipment or of a process to operate in a normal or usual manner so that emissions of arsenic are increased.

"Pot furnace" means a glass melting furnace that contains one or more refractory vessels in which glass is melted by indirect heating. The openings of the vessels are in the outside wall of the furnace and are covered with refractory stoppers during melting.

"Rebrick" means cold replacement of damaged or worn refractory parts of the glass melting furnace. Rebrick includes replacement of the refractories comprising the bottom, sidewalls, or roof of the melting vessel; replacement of refractory work in the heat exchanger; and replacement of refractory portions of the glass conditioning and distribution system.

"Shutdown" means the cessation of operation of an affected source for any purpose.

"Theoretical arsenic emissions factor" means the amount of inorganic arsenic, expressed in grams per kilogram of glass produced, as determined based on a material balance.

"Uncontrolled total arsenic emissions" means the total inorganic arsenic in the glass melting furnace exhaust gas preceding any add-on emission control device.

[51 FR 28025, Aug. 4, 1986; 51 FR 35355, Oct. 3, 1986]

§ 61.162 Emission limits.

(a) The owner or operator of an existing glass melting furnace subject to the provisions of this subpart shall comply with either paragraph (a)(1) or (a)(2) of this section; except as provided in paragraph (c) of this section.

(1) Uncontrolled total arsenic emissions from the glass melting furnace shall be less than 2.5 Mg per year, or

(2) Total arsenic emissions from the glass melting furnace shall be conveyed to a control device and reduced by at least 85 percent.

(b) The owner or operator of a new or modified glass melting furnace subject to the provisions of this subpart shall comply with either paragraph (b)(1) or (b)(2) of this section, except as provided in paragraph (c) of this section.

(1) Uncontrolled total arsenic emissions from the glass melting furnace shall be less than 0.4 Mg per year, or

(2) Total arsenic emissions from the glass melting furnace shall be conveyed to a control device and reduced by at least 85 percent.

(c) An owner or operator of a source subject to the requirements of this section may, after approval by the Administrator, bypass the control device to which arsenic emissions from the furnace are directed for a limited period of time for designated purposes such as maintenance of the control device, as specified in § 61.165(e).

(d) At all times, including periods of startup, shutdown, and malfunction, the owner or operator of a glass melting furnace subject to the provisions of this subpart shall operate and

maintain the furnace and associated air pollution control equipment in a manner consistent with good air pollution control practice for minimizing emissions of inorganic arsenic to the atmosphere to the maximum extent practicable. Determination of whether acceptable operating and maintenance procedures are being used will be based on information available to the Administrator, which may include, but is not limited to, monitoring results, review of operating and maintenance procedures, inspection of the source, and review of other records.

§ 61.163 Emission monitoring.

(a) An owner or operator of a glass melting furnace subject to the emission limit in § 61.162(a)(2) or § 61.162(b)(2) shall:

(1) Install, calibrate, maintain, and operate a continuous monitoring system for the measurement of the opacity of emissions discharged into the atmosphere from the control device; and

(2) Install, calibrate, maintain, and operate a monitoring device for the continuous measurement of the temperature of the gas entering the control device.

(b) All continuous monitoring systems and monitoring devices shall be installed and operational prior to performance of an emission test required by § 61.164(a). Verification of operational status shall, at a minimum, consist of an evaluation of the monitoring system in accordance with the requirements and procedures contained in Performance Specification 1 of Appendix B of 40 CFR part 60.

(c) During the emission test required in § 61.164(a) each owner or operator subject to paragraph (a) of this section shall:

(1) Conduct continuous opacity monitoring from the beginning of the first test run until the completion of the third test run. Process and control equipment shall be operated in a manner that will minimize opacity of emissions, subject to the Administrator's approval.

(2) Calculate 6-minute opacity averages from 24 or more data points equally spaced over each 6-minute period during the test runs.

(3) Determine, based on the 6-minute opacity averages, the opacity value corresponding to the 97.5 percent upper confidence level of a normal or lognormal (whichever the owner or operator determines is more representative) distribution of the average opacity values.

(4) Conduct continuous monitoring of the temperature of the gas entering the control device from the beginning of the first test run until completion of the third test run.

(5) Calculate 15-minute averages of the temperature of the gas entering the control device during each test run.

(d) An owner or operator may re-determine the values described in paragraph (c) of this section during any emission test that demonstrates compliance with the emission limits in § 61.162(a)(2) or § 61.162(b)(2).

(e) The requirements of § 60.13(d) and § 60.13(f) shall apply to an owner or operator subject to paragraph (a) of this section.

(f) Except for system breakdowns, repairs, calibration checks, and zero and span adjustments required under § 60.13(d), all continuous monitoring systems shall be in continuous operation and shall meet minimum frequency of operation requirements by completing a minimum of one cycle of sampling and analyzing for each successive 10-second period and one cycle of data recording for each successive 6-minute period.

(g) An owner or operator subject to paragraph (a) of this section shall:

(1) Reduce all opacity data to 6-minute averages. Six-minute averages shall be calculated from 24 or more data points equally spaced over each 6-minute period. Data recorded during periods of monitoring system breakdowns, repairs, calibration checks, and zero and span adjustments shall not be included in the data averages calculated under this paragraph, and

(2) Calculate 15-minute averages of the temperature of the gas entering the control device for each 15-minute operating period.

(h) After receipt and consideration of written application, the Administrator may approve alternative monitoring systems for the measurement of

one or more process or operating parameters that is or are demonstrated to enable accurate and representative monitoring of a properly operating control device. Upon approval of an alternative monitoring system for an affected source, the Administrator will specify requirements to replace the requirements of paragraphs (a)–(g) of this section for that system.

§ 61.164 Test methods and procedures.

(a) To demonstrate compliance with § 61.162, the owner or operator shall conduct emission tests, reduce test data, and follow the procedures specified in this section unless the Administrator:

(1) Specifies or approves, in specific cases, the use of a reference method with minor changes in methodology;

(2) Approves the use of an equivalent method;

(3) Approves the use of an alternative method the results of which he has determined to be adequate for indicating whether a specific source is in compliance; or

(4) Waives the requirement for emission tests as provided under § 61.13.

(b) Unless a waiver of emission testing is obtained, the owner or operator shall conduct emission tests required by this section:

(1) No later than 90 days after the effective date of this subpart for a source that has an initial startup date preceding the effective date; or

(2) No later than 90 days after startup for a source that has an initial startup date after the effective date.

(3) At such other times as may be required by the Administrator under section 112 of the Act.

While the source is operating under such conditions as the Administrator may specify, based on representative performance of the source.

(c) To demonstrate compliance with § 61.162(a)(1) when less than 8.0 Mg per year of elemental arsenic is added to any existing glass melting furnace, or to demonstrate compliance with § 61.162(b)(1) when less than 1.0 Mg per year of elemental arsenic is added to any new or modified glass melting furnace, an owner or operator shall:

(1) Derive a theoretical uncontrolled arsenic emission factor (T_i), in grams

of elemental arsenic per kilogram of glass produced, based on material balance calculations for each arsenic-containing glass type (i) produced during the 12-month period, as follows:

$$T_i = (A_{bi} \times W_{bi}) + (A_{ci} \times W_{ci}) - A_{di}$$

Where:

T_i = the theoretical uncontrolled arsenic emission factor (g/kg) for each glass type (i).

A_{bi} = fraction by weight of elemental arsenic in the fresh batch for each glass type (i).

W_{bi} = weight (g) of fresh batch melted per kg of glass produced for each glass type (i).

A_{ci} = fraction by weight of elemental arsenic in cullet for each glass type (i).

W_{ci} = weight (g) of cullet melted per kg of glass produced for each glass type (i).

A_{di} = weight (g) of elemental arsenic per kg glass produced for each glass type (i).

(2) Estimate theoretical uncontrolled arsenic emissions for the 12-month period for each arsenic-containing glass type as follows:

$$Y_i = \frac{(T_i \times G_i)}{10^6}$$

Where:

Y_i = the theoretical uncontrolled arsenic emission estimate for the 12-month period for each glass type (Mg/year).

T_i = the theoretical uncontrolled arsenic emission factor for each type of glass (i) produced during the 12-month period as calculated in paragraph (c)(1) of this section (g/kg).

G_i = the quantity (kg) of each arsenic-containing glass type (i) produced during the 12-month period.

(3) Estimate the total theoretical uncontrolled arsenic emissions for the 12-month period by finding the sum of the values calculated for Y_i in paragraph (c)(2) of this section.

(4) If the value determined in paragraph (c)(3) of this section is equal to or greater than the applicable limit in § 61.162(a)(1) or (b)(1), conduct the emission testing and calculations described in paragraphs (d)(1) through (d)(5) of this section. If the value is less than the applicable limit, the source is in compliance and no emis-

sion testing or additional calculations are required.

(d) To demonstrate compliance with § 61.162(a)(1) when 8.0 Mg per year or more of elemental arsenic are added to any existing glass melting furnace, or to demonstrate compliance with § 61.162(b)(1) when 1.0 Mg per year or more of elemental arsenic is added to any new or modified glass melting furnace, an owner or operator shall:

(1) Estimate the theoretical uncontrolled arsenic emissions for each glass type for the 12-month period by performing the calculations described in paragraphs (c)(1) and (c)(2) of this section.

(2) Conduct emission testing to determine the actual uncontrolled arsenic emission rate during production of the arsenic-containing glass type with the highest theoretical uncontrolled arsenic emissions as calculated under paragraph (d)(1) of this section. The owner or operator shall use the following test methods and procedures:

(i) Use Method 108 in Appendix B to this part for determining the arsenic emission rate (g/h). The emission rate shall equal the arithmetic mean of the results of three 60-minute test runs.

(ii) Use the following methods in Appendix A to 40 CFR part 60:

(A) Method 1 for sample and velocity traverse.

(B) Method 2 for velocity and volumetric flowrate.

(C) Method 3 for gas analysis.

(D) For sources equipped with positive pressure fabric filters, use Section 4 of Method 5D to determine a suitable sampling location and procedure.

(3) Determine the actual uncontrolled arsenic emission factor (R_a) in grams of elemental arsenic per kilogram of glass produced, as follows:

$$R_a = E_a \div P$$

Where:

R_a = the actual uncontrolled arsenic emission factor (g/kg).

E_a = the actual uncontrolled arsenic emission rate (g/h) from paragraph (d)(2) of this section.

P = the rate of glass production (kg/h), determined by dividing the weight (kg) of glass pulled from the furnace during the emission test by the number of hours (h) taken to perform the test under paragraph (d)(2) of this section.

(4) Calculate a correction factor to relate the theoretical and the actual uncontrolled arsenic emission factors as follows:

$$F = R_a \div T_i$$

Where:

F = the correction factor.

R_a = the actual uncontrolled arsenic emission factor (g/kg) determined in paragraph (d)(3) of this section.

T_i = the theoretical uncontrolled arsenic emission factor (g/kg) determined in paragraph (c)(1) of this section for the same glass type for which R_a was determined.

(5) Determine the uncontrolled arsenic emission rate for the 12-month period, as follows:

$$U = \frac{\sum_{i=1}^n (T_i \times F \times G_i)}{10^6}$$

Where:

U = the uncontrolled arsenic emission rate for the 12-month period (Mg/year).

T_i = the theoretical uncontrolled arsenic emission factor for each arsenic-containing glass type (i) produced during the 12-month period, as calculated in paragraph (c)(1) of this section (g/kg).

F = the correction factor calculated in paragraph (d)(4) of this section.

G_i = the quantity (kg) of each arsenic-containing glass type (i) produced during the 12-month period.

n = the number of arsenic-containing glass types produced during the 12-month period.

(6) If the value determined in paragraph (d)(5) of this section is less than the applicable limit in § 61.162(a)(1) or (b)(1), the source is in compliance.

(e) To demonstrate compliance with § 61.162(a)(2) or (b)(2), an owner or operator shall:

(1) Conduct emission testing to determine the percent reduction of inorganic arsenic emissions being achieved by the control device, using the following test methods and procedures:

(i) Use Method 108 in Appendix B to this part to determine the concentration of arsenic in the gas streams entering and exiting the control device. Conduct three 60-minute test runs, each consisting of simultaneous test:

ing of the inlet and outlet gas streams. The gas streams shall contain all the gas exhausted from the glass melting furnace.

(ii) Use the following methods in Appendix A to 40 CFR part 60:

(A) Method 1 for sample and velocity traverses.

(B) Method 2 for velocity and volumetric flowrate.

(C) Method 3 for gas analysis.

(D) For sources equipped with positive pressure fabric filters, use Section 4 of Method 5D to determine a suitable sampling location and procedure.

(2) Calculate the percent emission reduction for each run as follows:

$$D = \frac{(C_a - C_c) \times 100}{C_b}$$

Where:

D = the percent emission reduction.

C_b = the arsenic concentration of the stack gas entering the control device, as measured by Method 108.

C_a = the arsenic concentration of the stack gas exiting the control device, as measured by Method 108.

(3) Determine the average percent reduction of arsenic by calculating the arithmetic mean of the results for the three runs. If it is at least 85 percent, the source is in compliance.

[51 FR 28025, Aug. 4, 1986; 51 FR 35355, Oct. 3, 1986, as amended at 55 FR 22027, May 31, 1990]

§ 61.165 Reporting and recordkeeping requirements.

(a) Each owner or operator of a source subject to the requirements of § 61.162 shall maintain at the source for a period of at least 2 years and make available to the Administrator upon request a file of the following records:

(1) All measurements, including continuous monitoring for measurement of opacity, and temperature of gas entering a control device;

(2) Records of emission test data and all calculations used to produce the required reports of emission estimates to demonstrate compliance with § 61.162;

(3) All continuous monitoring system performance evaluations, including calibration checks and adjustments;

(4) The occurrence and duration of all startups, shutdowns, and malfunctions of the furnace;

(5) All malfunctions of the air pollution control system;

(6) All periods during which any continuous monitoring system or monitoring device is inoperative;

(7) all records of maintenance and repairs for each air pollution control system, continuous monitoring system, or monitoring device;

(b) Each owner or operator who is given approval by the Administrator to bypass a control device under paragraph (e) of this section shall maintain at the source for a period of at least 2 years and make available to the Administrator upon request a file of the following records:

(1) The dates the control device is bypassed; and

(2) Steps taken to minimize arsenic emissions during the period the control device was bypassed.

(c) Each owner or operator of a source subject to the emission limit in § 61.162(a)(1) or (b)(1) shall determine and record at the end of every 6 months the uncontrolled arsenic emission rate for the preceding and forthcoming 12-month periods. The determinations shall:

(1) Be made by following the procedures in § 61.164(c)(1), (c)(2), and (c)(3); or in § 61.164(d)(5), whichever is applicable; and

(2) Take into account changes in production rates, types of glass produced, and other factors that would affect the uncontrolled arsenic emission rate.

(d) Each owner or operator of a source subject to the provisions of this subpart shall:

(1) Provide the Administrator 30 days prior notice of any emission test required in § 61.164 to afford the Administrator the opportunity to have an observer present; and

(2) Submit to the Administrator a written report of the results of the emission test and associated calculations required in § 61.164(d) or (e), as applicable, within 60 days after conducting the test.

(3) Submit to the Administrator a written report of the arsenic emission estimates calculated under § 61.164(c):

(i) Within 45 days after the effective date of this subpart for a source that has an initial startup date preceding the effective date; or

(ii) Within 45 days after startup for a source that has an initial startup date after the effective date.

(4) Submit to the Administrator a written report of the uncontrolled arsenic emission rates determined in accordance with paragraph (c) of this section, if:

(i) The emission rate for the preceding 12-month period (or preceding 6-month period for the first 6-month determination) exceeded the applicable limit in § 61.162(a)(1) or (b)(1).

(ii) The emission rate for the forthcoming 12-month period will exceed the applicable limit in § 61.162(a)(1) or (b)(1). In this case, the owner or operator shall also notify the Administrator of the anticipated date of the emission test to demonstrate compliance with the applicable limit in § 61.162(a)(2) or (b)(2).

(5) Ensure that the reports required in paragraph (d)(4) of this section are postmarked by the tenth day following the end of the 6-month reporting period.

(e) To obtain approval to bypass a control device, as provided in § 61.162(c), an owner or operator of a source subject to this subpart may make written application to the Administrator. Each application for such a waiver shall be submitted to the Administrator no later than 60 days before the bypass period would begin and shall include:

(1) Name and address of the owner or operator;

(2) Location of the source;

(3) A brief description of the nature, size, design, and method of operation of the source;

(4) The reason it is necessary to bypass the control device;

(5) The length of time it will be necessary to by-pass the control device;

(6) Steps that will be taken to minimize arsenic emissions during the period the control device will be bypassed.

(7) The quantity of emissions that would be released while the control device is by-passed if no steps were taken to minimize emissions;

(8) The expected reduction in emissions during the by-pass period due to the steps taken to minimize emissions during this period; and

(9) The type of glass to be produced during the bypass period, and, if applicable, an explanation of why non-arsenic or lower-arsenic-containing glass cannot be melted in the furnace during the bypass period.

(f) Each owner or operator required to install and operate a continuous opacity monitoring system under § 61.163 shall:

(1) Submit a written report to the Administrator of the results of the continuous monitoring system evaluation required under § 61.163(b) within 60 days after conducting the evaluation.

(2) Submit a written report to the Administrator every 6 months if excess opacity occurred during the preceding 6-month period. For purposes of this paragraph, an occurrence of excess opacity is any 6-minute period during which the average opacity, as measured by the continuous monitoring system, exceeds the opacity level determined under § 61.163(c)(3) or the opacity level re-determined under § 61.163(d).

(3) Ensure that any semiannual report of excess opacity required by paragraph (f)(2) of this section is postmarked by the thirtieth day following the end of the 6-month period and includes the following information:

(i) The magnitude of excess opacity, any conversion factor(s) used, and the date and time of commencement and completion of each occurrence of excess opacity.

(ii) Specific identification of each occurrence of excess opacity that occurs during startups, shutdowns, and malfunctions of the source.

(iii) The date and time identifying each period during which the continuous monitoring system was inoperative, except for zero and span checks, and the nature of the system repairs or adjustments.

(Approved by the Office of Management and Budget under control number 2060-0043)

Subpart O—National Emission Standard for Inorganic Arsenic Emissions from Primary Copper Smelters

SOURCE: 51 FR 28029, Aug. 4, 1986, unless otherwise noted.

§ 61.170 Applicability and designation of source.

The provisions of this subpart are applicable to each copper converter at any new or existing primary copper smelter, except as noted in § 61.172(a).

§ 61.171 Definitions.

All terms used in this subpart shall have the meanings given to them in the Act, in subpart A of part 61, and in this section as follows:

"Blowing" means the injection of air or oxygen-enriched air into a molten converter bath.

"Charging" means the addition of a molten or solid material to a copper converter.

"Control device" means the air pollution control equipment used to collect particulate matter emissions.

"Converter arsenic charging rate" means the hourly rate at which arsenic is charged to the copper converters in the copper converter department based on the arsenic content of the copper matte and of any lead matte that is charged to the copper converters.

"Copper converter" means any vessel in which copper matte is charged and is oxidized to copper.

"Copper converter department" means all copper converters at a primary copper smelter.

"Copper matte" means any molten solution of copper and iron sulfides produced by smelting copper sulfide ore concentrates or calcines.

"Holding of a copper converter" means suspending blowing operations while maintaining in a heated state the molten bath in the copper converter.

"Inorganic arsenic" means the oxides and other noncarbon compounds of the element arsenic includ-

ed in particulate matter, vapors, and aerosols.

"Lead matte" means any molten solution of copper and other metal sulfides produced by reduction of sinter product from the oxidation of lead sulfide ore concentrates.

"Malfunction" means any sudden failure of air pollution control equipment or process equipment or of a process to operate in a normal or usual manner so that emissions of inorganic arsenic are increased.

"Opacity" means the degree to which emissions reduce the transmission of light.

"Particulate matter" means any finely divided solid or liquid material, other than uncombined water, as measured by the specified reference method.

"Pouring" means the removal of blister copper from the copper converter bath.

"Primary copper smelter" means any installation or intermediate process engaged in the production of copper from copper-bearing materials through the use of pyrometallurgical techniques.

"Primary emission control system" means the hoods, ducts, and control devices used to capture, convey, and collect process emissions.

"Process emissions" means inorganic arsenic emissions from copper converters that are captured directly at the source of generation.

"Secondary emissions" means inorganic arsenic emissions that escape capture by a primary emission control system.

"Secondary hood system" means the equipment (including hoods, ducts, fans, and dampers) used to capture and transport secondary inorganic arsenic emissions.

"Shutdown" means the cessation of operation of a stationary source for any reason.

"Skimming" means the removal of slag from the molten converter bath.

§ 61.172 Standard for new and existing sources.

(a) The provisions of paragraphs (b)-(f) of this section do not apply to any copper converter at a facility

where the total arsenic charging rate for the copper converter department averaged over a 1-year period is less than 75 kg/h, as determined under § 61.174(f).

(b) The owner or operator of each copper converter subject to the provisions of this subpart shall reduce inorganic arsenic emissions to the atmosphere by meeting the following design, equipment, work practice, and operational requirements:

(1) Install, operate, and maintain a secondary hood system on each copper converter. Each secondary hood system shall consist of a hood enclosure, air curtain fan(s), exhaust system fan(s), and ductwork that conveys the captured emissions to a control device, and shall meet the following specifications:

(i) The configuration and dimensions of the hood enclosure shall be such that the copper converter mouth, charging ladles, skimming ladles, and any other material transfer vessels used will be housed within the confines or influence of the hood enclosure during each mode of copper converter operation.

(ii) The back of the hood enclosure shall be fully enclosed and sealed against the primary hood. Portions of the side-walls in contact with the copper converter shall be sealed against the converter.

(iii) Openings in the top and front of the hood enclosure to allow for the entry and egress of ladles and crane apparatus shall be minimized to the fullest extent practicable.

(iv) The hood enclosure shall be fabricated in such a manner and of materials of sufficient strength to withstand incidental contact with ladles and crane apparatus with no significant damage.

(v) One side-wall of the hood enclosure shall be equipped with a horizontal-slotted plenum along the top, and the opposite side-wall shall be equipped with an exhaust hood. The horizontal-slotted plenum shall be designed to allow the distance from the base to the top of the horizontal slot to be adjustable up to a dimension of 76 mm.

(vi) The horizontal-slotted plenum shall be connected to a fan. When acti-

vated, the fan shall push air through the horizontal slot, producing a horizontal air curtain above the copper converter that is directed to the exhaust hood. The fan power output installed shall be sufficient to overcome static pressure losses through the ductwork upstream of the horizontal-slotted plenum and across the plenum, and to deliver at least 22,370 watts (30 air horsepower) at the horizontal-slotted plenum discharge.

(vii) The exhaust hood shall be sized to completely intercept the airstream from the horizontal-slotted plenum combined with the additional airflow resulting from entrainment of the surrounding air. The exhaust hood shall be connected to a fan. When activated, the fan shall pull the combined airstream into the exhaust hood.

(viii) The entire secondary hood system shall be equipped with dampers and instrumentation, as appropriate, so that the desired air curtain and exhaust flow are maintained during each mode of copper converter operation.

(2) Optimize the capture of secondary inorganic arsenic emissions by operating the copper converter and secondary hood system at all times as follows:

(i) Copper converter. (A) Increase the air curtain and exhaust flow rates to their optimum conditions prior to raising the primary hood and rolling the copper converter out for charging, skimming, or pouring.

(B) Once rolled out, prior to the commencement of skimming or pouring, hold the copper converter in an idle position until fuming from the molten bath has been minimized.

(C) During skimming, raise the receiving ladle off the ground and position the ladle as close to the copper converter mouth as possible to minimize the drop distance between the converter mouth and the receiving ladle.

(D) Control the rate of flow into the receiving ladle to the extent practicable to minimize fuming.

(E) Upon the completion of each charge, withdraw the charging ladle or vessel used from the confines of the secondary hood in a slow, deliberate manner.

(F) During charging, skimming, or pouring, ensure that the crane block does not disturb the air flow between the horizontal-slotted plenum and the exhaust hood.

(ii) Secondary hood system. (A) Operate the secondary hood system under conditions that will result in the maximum capture of inorganic arsenic emissions.

(B) Within 30 days after the effective date of this subpart, or within 30 days after the initial operation of each secondary hood system, whichever comes later, provide to the Administrator a list of operating conditions for the secondary hood system that will result in the maximum capture of inorganic arsenic emissions. This list shall specify the operating parameters for the following:

(1) The dimensions of the horizontal slot.

(2) The velocity of air through the horizontal slot during each mode of converter operation.

(3) The distance from the horizontal slot to the exhaust hood.

(4) The face velocity at the opening of the exhaust hood during each mode of converter operation.

(C) Operate the secondary hood system under the conditions listed in paragraph (b)(2)(ii)(B) of this section, unless otherwise specified by the Administrator.

(D) Notify the Administrator in writing within 30 days if there is any change in the operating conditions submitted pursuant to the requirements of paragraph (b)(2)(ii)(B) that will result in any reduction in the maximum capture of inorganic arsenic emissions.

(3) Comply with the following inspection and maintenance requirements after installing the secondary hood system required in paragraph (b)(1) of this section:

(i) At least once every month, visually inspect the components of the secondary hood system that are exposed to potential damage from crane and ladle operation, including the hood enclosure, side- and back-wall hood seals, and the horizontal slot.

(ii) Replace or repair any defective or damaged components of the secondary hood system within 30 days after

discovering the defective or damaged components.

(c) No owner or operator of a copper converter subject to the provisions of this subpart shall cause or allow to be discharged into the atmosphere any copper converter secondary emissions that exit from a control device and contain particulate matter in excess of 11.6 milligrams per dry standard cubic meter.

(d) The owner or operator of a copper converter subject to the provisions of this subpart shall submit a description of a plan for control of inorganic arsenic emissions from the copper converter and associated air pollution control equipment. This plan shall be submitted within 90 days after the effective date of this subpart, unless a waiver of compliance is granted under § 61.11. If a waiver of compliance is granted, the plan shall be submitted on a date set by the Administrator. Approval of the plan will be granted by the Administrator provided he finds that:

(1) It includes a systematic procedure for identifying malfunctions and for reporting them immediately to smelter supervisory personnel.

(2) It specifies the procedures that will be followed to ensure that equipment or process breakdowns due entirely or in part to poor maintenance or other preventable conditions do not occur.

(3) It specifies the measures that will be taken to ensure compliance with paragraph (b)(2) of this section.

(e) The owner or operator shall implement the plan required under paragraph (d) of this section unless otherwise specified by the Administrator.

(f) At all times, including periods of startup, shutdown, and malfunction, the owner or operator of a copper converter subject to the provisions of this subpart shall operate and maintain the converter and associated air pollution control equipment in a manner consistent with good air pollution control practice for minimizing emissions of inorganic arsenic to the atmosphere to the maximum extent practicable. Determination of whether acceptable operating and maintenance procedures are being used will be based on information available to the Administrator,

which may include, but is not limited to, monitoring results, review of operating and maintenance procedures, inspection of the source, and review of other records.

§ 61.173 Compliance provisions.

(a) The owner or operator of each copper converter to which § 61.172(b)-(f) applies shall demonstrate compliance with the requirements of § 61.172(b)(1) as follows:

(1) The owner or operator of each existing copper converter shall install a secondary hood system to meet the requirements of § 61.172(b)(1) no later than 90 days after the effective date, unless a waiver of compliance has been approved by the Administrator in accordance with § 61.11.

(2) The owner or operator of each new copper converter shall install a secondary hood system to meet the requirements of § 61.172(b)(1) prior to the initial startup of the converter, except that if startup occurs prior to the effective date, the owner or operator shall meet the requirements of § 61.172(b)(1) on the effective date.

§ 61.174 Test methods and procedures.

(a) To determine compliance with § 61.172(c), the owner or operator shall conduct emission tests and reduce the test data in accordance with the test methods and procedures contained in this section unless the Administrator:

(1) Specifies or approves, in specific cases, the use of a reference method with minor changes in methodology,

(2) Approves the use of an equivalent method,

(3) Approves the use of an alternative method, the results of which he has determined to be adequate for indicating whether a specific source is in compliance, or

(4) Waives the requirement for emission tests as provided in § 61.13.

(b) The owner or operator shall conduct the emission tests required in paragraph (a) of this section:

(1) After achieving the optimum operating conditions submitted under § 60.172(b)(2)(ii)(B) for the equipment required in § 61.172(b)(1), but no later than 90 days after the effective date of this subpart in the case of an existing copper converter or a copper con-

verter that has an initial startup date preceding the effective date, or

(2) After achieving the optimum operating conditions submitted under § 60.172(b)(2)(ii)(B) for the equipment required in § 61.172(b)(1), but no later than 90 days after startup in the case of a new copper converter, initial startup of which occurs after the effective date, or

(3) At such other times as may be required by the Administrator under section 114 of the Act.

(c) The owner or operator shall conduct each emission test under representative operating conditions and at sample locations subject to the Administrator's approval, and shall make available to the Administrator such records as may be necessary to determine the conditions of the emission test.

(d) For the purpose of determining compliance with § 61.172(c), the owner or operator shall use reference methods in 40 CFR part 60, Appendix A, as follows:

(1) Method 5 for the measurement of particulate matter,

(2) Method 1 for sample and velocity traverses,

(3) Method 2 for velocity and volumetric flow rate,

(4) Method 3 for gas analysis, and

(5) Method 4 for stack gas moisture.

(e) For Method 5, the sampling time for each run shall be at least 60 minutes and the minimum sampling volume shall be 0.85 dscm (30 dscf) except that smaller times or volumes when necessitated by process variables or other factors may be approved by the Administrator.

(f) For the purpose of determining applicability under § 61.172(a), the owner or operator shall determine the converter arsenic charging rate as follows:

(1) Collect daily grab samples of copper matte and any lead matte charged to the copper converters.

(2) Each calendar month, from the daily grab samples collected under paragraph (f)(1) of this section, put together a composite copper matte sample and a composite lead matte sample. Analyze the composite samples individually using Method 108A, 108B, or 108C to determine the weight

percent of inorganic arsenic contained in each sample.

(3) Calculate the converter arsenic charging rate once per month using the following equation:

$$R_c = \frac{\sum_{i=1}^n \frac{A_c W_{ci} + A_l W_{li}}{100 H_i}}$$

Where:

R_c is the converter arsenic charging rate (kg/h).

A_c is the monthly average weight percent of arsenic in the copper matte charged during the month (%) as determined under paragraph (f)(2) of this section.

A_l is the monthly average weight percent of arsenic in the lead matte charged during the month (%) as determined under paragraph (f)(2) of this section.

W_{ci} is the total weight of copper matte charged to a copper converter during the month (kg).

W_{li} is the total weight of lead matte charged to a copper converter during the month (kg).

H_i is the total number of hours the copper converter department was in operation during the month (h).

n is the number of copper converters in operation during the month.

(4) Determine an annual arsenic charging rate for the copper converter department once per month by computing the arithmetic average of the 12 monthly converter arsenic charging rate values (R_c) for the preceding 12-month period.

(g) An owner or operator may petition the Administrator for a modified sampling and analysis schedule if analyses performed for the first 12-month period after the effective date show the source to be considerably below the applicability limit prescribed in § 61.172(a).

[51 FR 28029, Aug. 4, 1986, as amended at 55 FR 22027, May 31, 1990]

§ 61.175 Monitoring requirements.

(a) Each owner or operator of a source that is subject to the emission limit specified in § 61.172(c) shall install, calibrate, maintain, and operate a continuous monitoring system for the measurement of the opacity of emissions discharged from the control device according to the following procedures:

(1) Ensure that each system is installed and operational no later than 90 days after the effective date of this subpart for a source that has an initial startup date preceding the effective date; and no later than 90 days after startup for other sources. Verification of the operational status shall, as a minimum, consist of an evaluation of the monitoring system in accordance with the requirements and procedures contained in Performance Specification 1 of Appendix B of 40 CFR part 60.

(2) Comply with the provisions of § 60.13(d) of 40 CFR part 60.

(3) Except for system breakdowns, repairs, calibration checks, and zero span adjustments, ensure that each continuous monitoring system is in continuous operation and meets frequency of operation requirements by completing a minimum of one cycle of sampling and analysis for each successive 10-second period and one cycle of data recording for each successive 6-minute period. Each data point shall represent the opacity measured for one cycle of sampling and analysis and shall be expressed as percent opacity.

(b) Except as required in paragraph (c) of this section, calculate 1-hour opacity averages from 360 or more consecutive data points equally spaced over each 1-hour period. Data recorded during periods of monitoring system breakdowns, repairs, calibration checks, and zero and span adjustments shall not be included in the data averages computed under this paragraph.

(c) No later than 60 days after each continuous opacity monitoring system required in paragraph (a) of this section becomes operational, the owner or operator shall establish a reference opacity level for each monitored emission stream according to the following procedures:

(1) Conduct continuous opacity monitoring over a preplanned period of not less than 36 hours during which the processes and emission control equipment upstream of the monitoring system are operating under representative operating conditions subject to the Administrator's approval. This period shall include the time during

which the emission test required by § 61.13 is conducted.

(2) Calculate 6-minute averages of the opacity readings using 36 or more consecutive data points equally spaced over each 6-minute period.

(3) Calculate 1-hour average opacity values using 10 successive 6-minute average opacity values (i.e., calculate a new 1-hour average opacity value every 6 minutes). Determine the highest 1-hour average opacity value observed during the 36-hour preplanned test period.

(4) Calculate the reference opacity level by adding 5 percent opacity to the highest 1-hour average opacity calculated in paragraph (c)(3) of this section.

(d) The owner or operator may redetermine the reference opacity level for the copper converter secondary emission stream at the time of each emission test that demonstrates compliance with the emission limit required in § 61.172(c) according to the provisions of paragraphs (c)(1) through (c)(4) of this section.

(e) With a minimum of 30 days prior notice, the Administrator may require the owner or operator to redetermine the reference opacity level for any monitored emission stream.

(f) Each owner or operator who is required to install the equipment specified in § 61.172(b)(1) for the capture of secondary copper converter emissions shall install, calibrate, maintain, and operate a continuous monitoring device on each secondary hood system for the measurement of the air flow through the horizontal-slotted plenum and through the exhaust hood. Each device shall be installed and operational no later than 90 days after the effective date of this subpart for a source that has an initial startup preceding the effective date; and no later than 90 days after startup for other sources.

(g) Each owner or operator subject to the requirements in paragraph (f) of this section shall establish for each secondary hood system reference air flow rates for the horizontal-slotted plenum and exhaust hood for each mode of converter operation. The reference flow rates shall be established when the equipment is operating

under the optimum operating conditions required in § 61.172(b)(2)(ii).

(h) Each owner or operator shall install the continuous monitoring systems and monitoring devices required in paragraphs (a) and (f) of this section in such a manner that representative measurements of emissions and process parameters are obtained.

§ 61.176 Recordkeeping requirements.

(a) Each owner or operator subject to the requirements of § 61.172(b)(1) shall maintain at the source for a period of at least 2 years records of the visual inspections, maintenance, and repairs performed on each secondary hood system as required in § 61.172(b)(3).

(b) Each owner or operator subject to the provisions of § 61.172(c) shall maintain at the source for a period of at least 2 years and make available to the Administrator upon request a file of the following records:

(1) All measurements, including continuous monitoring for measurement of opacity;

(2) Records of emission test data and all calculations used to produce the required reports of emission estimates to demonstrate compliance with § 61.172(c);

(3) All continuous monitoring system performance evaluations, including calibration checks and adjustments;

(4) The occurrence and duration of all startups, shutdowns, and malfunctions of the copper converters;

(5) All malfunctions of the air pollution control system;

(6) All periods during which any continuous monitoring system or device is inoperative;

(7) All maintenance and repairs performed on each air pollution control system, continuous monitoring system, or monitoring device;

(8) All records of 1-hour average opacity levels for each separate control device; and

(9) For each secondary hood system:

(i) The reference flow rates for the horizontal-slotted plenum and exhaust hood for each converter operating mode established under § 61.175(g);

(ii) The actual flow rates; and

(iii) A daily log of the start time and duration of each converter operating mode.

(c) Each owner or operator subject to the provisions of this subpart shall maintain at the source for a period of at least 2 years and make available to the Administrator upon request the following records:

(1) For each copper converter, a daily record of the amount of copper matte and lead matte charged to the copper converter and the total hours of operation.

(2) For each copper converter department, a monthly record of the weight percent of arsenic contained in the copper matte and lead matte as determined under § 61.174(f).

(3) For each copper converter department, the monthly calculations of the average annual arsenic charging rate for the preceding 12-month period as determined under § 61.174(f).

(Approved by the Office of Management and Budget under control number 2060-0044)

§ 61.177 Reporting requirements.

(a) Each owner or operator subject to the provisions of § 61.172(c) shall:

(1) Provide the Administrator 30 days prior notice of the emission test required in § 61.174(a) to afford the Administrator the opportunity to have an observer present; and

(2) Submit to the Administrator a written report of the results of the emission test required in § 61.174(a) within 60 days after conducting the test.

(b) Each owner or operator subject to the provisions of § 61.175(a) shall provide the Administrator at least 30 days prior notice of each reference opacity level determination required in § 61.175(c) to afford the Administrator the opportunity to have an observer present.

(c) Each owner or operator subject to the provisions of § 61.175(a) shall submit to the Administrator:

(1) Within 60 days after conducting the evaluation required in § 61.175(a)(1), a written report of the continuous monitoring system evaluation;

(2) Within 30 days after establishing the reference opacity level required in

§ 61.175(c), a written report of the reference opacity level. The report shall also include the opacity data used and the calculations performed to determine the reference opacity level, and sufficient documentation to show that process and emission control equipment were operating normally during the reference opacity level determination; and

(3) A written report each quarter of each occurrence of excess opacity during the quarter. For purposes of this paragraph, an occurrence of excess opacity is any 1-hour period during which the average opacity, as measured by the continuous monitoring system, exceeds the reference opacity level established under § 61.175(c).

(d) The owner or operator subject to the provisions of § 61.175(g) shall submit to the Administrator:

(1) A written report of the reference air flow rate within 30 days after establishing the reference air flow rates required in § 61.175(g);

(2) A written report each quarter of all air flow rates monitored during the preceding 3-month period that are less than 80 percent of the corresponding reference flow rate established for each converter operating mode; and

(3) A written report each quarter of any changes in the operating conditions of the emission capture system, emission control device, or the building housing the converters that might increase fugitive emissions.

(e) All quarterly reports shall be postmarked by the 30th day following the end of each 3-month period and shall include the following information:

(1) The magnitude of each occurrence of excess opacity, any conversion factor(s) used, and the dates and times of commencement and completion of each occurrence of excess opacity, the cause of each exceedance of the reference opacity level, and the measures taken to minimize emissions.

(2) The magnitude of each occurrence of reduced flow rate and the date and time of commencement and completion of each occurrence of reduced flow rate, the cause of the reduced flow rate, and the associated converter operating mode.

(3) Specific identification of each occurrence of excess opacity or reduced flow rate that occurs during startups, shutdowns, and malfunctions of the source.

(4) The date and time identifying each period during which the continuous monitoring system or monitoring device was inoperative, except for zero and span checks, and the nature of the system repairs or adjustments.

(5) Specific identification of each change in operating conditions of the emission capture system or control device, or in the condition of the building housing the converters.

(f) Each owner or operator of a source subject to the provisions of this subpart shall submit annually a written report to the Administrator that includes the monthly computations of the average annual converter arsenic charging rate as calculated under § 61.174(f)(4). The annual report shall be postmarked by the 30th day following the end of each calendar year.

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Subpart P—National Emission Standard for Inorganic Arsenic Emissions From Arsenic Trioxide and Metallic Arsenic Production Facilities

SOURCE: 51 FR 28033, Aug. 4, 1986, unless otherwise noted.

§ 61.180 Applicability and designation of sources.

The provisions of this subpart are applicable to each metallic arsenic production plant and to each arsenic trioxide plant that processes low-grade arsenic bearing materials by a roasting condensation process.

§ 61.181 Definitions.

All terms used in this subpart shall have the meanings given them in the Act, in subpart A of part 61, and in this section as follows:

Arsenic kitchen means a baffled brick chamber where inorganic arsenic vapors are cooled, condensed, and removed in a solid form.

Control device means the air pollution control equipment used to collect particulate matter emissions.

Curtail means to cease operations to the extent technically feasible to reduce emissions.

Inorganic arsenic means the oxides and other noncarbon compounds of the element arsenic included in particulate matter, vapors, and aerosols.

Malfunction means any sudden failure of air pollution control equipment or process equipment or of a process to operate in a normal or usual manner so that emissions of inorganic arsenic are increased.

Opacity means the degree to which emissions reduce the transmission of light.

Primary emission control system means the hoods, enclosures, ducts, and control devices used to capture, convey, and remove particulate matter from exhaust gases which are captured directly at the source of generation.

Process emissions means inorganic arsenic emissions that are captured and collected in a primary emission control system.

Roasting means the use of a furnace to heat arsenic plant feed material for the purpose of eliminating a significant portion of the volatile materials contained in the feed.

Secondary emissions means inorganic arsenic emissions that escape capture by a primary emission control system.

Shutdown means the cessation of operation of a stationary source for any purpose.

[51 FR 28033, Aug. 4, 1986; 51 FR 35355, Oct. 3, 1986]

§ 61.182 Standard for new and existing sources.

(a) Within 30 days after the effective date of this subpart, the owner or operator of each source to which this subpart applies shall identify and submit to the Administrator a list of potential sources (equipment and operations) of inorganic arsenic emissions.

(b) The owner or operator shall submit a description of an inspection, maintenance, and housekeeping plan

for control of inorganic arsenic emissions from the potential sources identified under paragraph (a) of this section. This plan shall be submitted within 90 days after the effective date of this subpart, unless a waiver of compliance is granted under § 61.11. If a waiver of compliance is granted, the plan shall be submitted on a date set by the Administrator. Approval of the plan will be granted by the Administrator provided he finds that:

(1) It achieves the following objectives in a manner that does not cause adverse impacts in other environmental media:

(i) Clean-up and proper disposal, wet-down, or chemical stabilization to the extent practicable (considering access and safety) of any dry, dusty material having an inorganic arsenic content greater than 2 percent that accumulates on any surface within the plant boundaries outside of a dust-tight enclosure.

(ii) Immediate clean-up and proper disposal, wet-down, or chemical stabilization of spills of all dry, dusty material having an inorganic arsenic content greater than 2 percent.

(iii) Minimization of emissions of inorganic arsenic to the atmosphere during removal of inorganic arsenic from the arsenic kitchen and from flue pulling operations by properly handling, wetting down, or chemically stabilizing all dusts and materials handled in these operations.

(2) It includes an inspection program that requires all process, conveying, and air pollution control equipment to be inspected at least once per shift to ensure that the equipment is being properly operated and maintained. The program will specify the evaluation criteria and will use a standardized checklist, which will be included as part of the plan required in paragraph (b) of this section, to document the inspection, maintenance, and housekeeping status of the equipment and that the objectives of paragraph (b)(1) of this section are being achieved.

(3) It includes a systematic procedure for identifying malfunctions and for reporting them immediately to supervisory personnel.

(4) It specifies the procedures that will be followed to ensure that equipment or process malfunctions due entirely or in part to poor maintenance or other preventable conditions do not occur.

(5) It includes a program for curtailing all operations necessary to minimize any increase in emissions of inorganic arsenic to the atmosphere resulting from a malfunction. The program will describe:

(i) The specific steps that will be taken to curtail each operation as soon as technically feasible after the malfunction is discovered.

(ii) The minimum time required to curtail each operation.

(iii) The procedures that will be used to ensure that the curtailment continues until after the malfunction is corrected.

(c) The owner or operator shall implement the plan required in paragraph (b) of this section until otherwise specified by the Administrator.

(d) At all times, including periods of startup, shutdown, and malfunction, the owner or operator of each source to which this subpart applies shall operate and maintain the source including associated air pollution control equipment in a manner consistent with good air pollution control practice for minimizing emissions of inorganic arsenic to the atmosphere to the maximum extent practicable. Determination of whether acceptable operating and maintenance procedures are being used will be based on information available to the Administrator, which may include, but is not limited to, monitoring results, review of operating and maintenance procedures, inspection of the source, and review of other records.

§ 61.183 Emission monitoring.

(a) The owner or operator of each source subject to the provisions of this subpart shall install, calibrate, maintain, and operate a continuous monitoring system for the measurement of the opacity of each arsenic trioxide and metallic arsenic process emission stream that exits from a control device.

(b) The owner or operator shall install, operate, and maintain each continuous monitoring system for the measurement of opacity required in paragraph (a) of this section according to the following procedures:

(1) Ensure that each system is installed and operational no later than 90 days after the effective date of this subpart for an existing source or a new source that has an initial startup date preceding the effective date. For a new source whose initial startup occurs after the effective date of this subpart, ensure that the system is installed and operational no later than 90 days after startup. Verification of the operational status shall, as a minimum, consist of an evaluation of the monitoring system in accordance with the requirements and procedures contained in Performance Specification 1 of Appendix B of 40 CFR part 60.

(2) Comply with the provisions of § 60.13(d) of 40 CFR part 60.

(3) Except for system breakdowns, repairs, calibration checks, and zero and span adjustments required under § 60.13(d), ensure that each continuous monitoring system is in continuous operation and meets frequency of operation requirements by completing a minimum of one cycle of sampling and analysis for each successive 10-second period and one cycle of data recording for each successive 6-minute period. Each data point shall represent the opacity measured for one cycle of sampling and analysis and shall be expressed as percent opacity.

(c) The owner or operator shall calculate 6-minute opacity averages from 36 or more consecutive data points equally spaced over each 6-minute period. Data recorded during periods of monitoring system breakdowns, repairs, calibration checks, and zero and span adjustments shall not be included in the data averages computed under this paragraph.

(d) No later than 60 days after each continuous opacity monitoring system required in paragraph (a) of this section becomes operational, the owner or operator shall establish a reference opacity level for each monitored emission stream according to the following procedures:

(1) Conduct continuous opacity monitoring over a preplanned period of not less than 36 hours during which the processes and emission control equipment upstream of the monitoring system are operating in a manner that will minimize opacity under representative operating conditions subject to the Administrator's approval.

(2) Calculate 6-minute averages of the opacity readings using 36 or more consecutive data points equally spaced over each 6-minute period.

(3) Establish the reference opacity level by determining the highest 6-minute average opacity calculated under paragraph (d)(2) of this section.

(e) With a minimum of 30 days prior notice, the Administrator may require an owner or operator to redetermine the reference opacity level for any monitored emission stream.

(f) Each owner or operator shall install all continuous monitoring systems or monitoring devices required in paragraph (a) of this section in such a manner that representative measurements of emissions or process parameters are obtained.

§ 61.184 Ambient air monitoring for inorganic arsenic.

(a) The owner or operator of each source to which this subpart applies shall operate a continuous monitoring system for the measurement of inorganic arsenic concentrations in the ambient air.

(b) The ambient air monitors shall be located at sites to detect maximum concentrations of inorganic arsenic in the ambient air in accordance with a plan approved by the Administrator that shall include the sampling and analytical method used.

(c) The owner or operator shall submit a written plan describing, and explaining the basis for, the design and adequacy of the monitoring network, sampling and analytical procedures, and quality assurance within 45 days after the effective date of this subpart.

(d) Each monitor shall be operated continuously except for a reasonable time allowance for instrument maintenance and calibration, for changing

filters, or for replacement of equipment needing major repair.

(e) Filters shall be changed daily and shall be analyzed and concentrations calculated within 30 days after filters are collected.

(f) The Administrator at any time may require changes in, or expansion of, the sampling program, including sampling and analytical protocols and network design.

§ 61.185 Recordkeeping requirements.

(a) Each owner or operator of a source subject to the provisions of this subpart shall maintain at the source for a period of at least 2 years the following records: All measurements, including continuous monitoring for measurement of opacity; all continuous monitoring system performance evaluations, including calibration checks and adjustments; all periods during which the continuous monitoring system or monitoring device is inoperative; and all maintenance and repairs made to the continuous monitoring system or monitoring device.

(b) Each owner or operator shall maintain at the source for a period of at least 2 years a log for each plant department in which the operating status of process, conveying, and emission control equipment is described for each shift. For malfunctions and upsets, the following information shall be recorded in the log:

- (1) The time of discovery.
- (2) A description of the malfunction or upset.
- (3) The time corrective action was initiated.
- (4) A description of corrective action taken.
- (5) The time corrective action was completed.
- (6) A description of steps taken to reduce emissions of inorganic arsenic to the atmosphere between the time of discovery and the time corrective action was taken.

(c) Each owner or operator subject to the provisions of this subpart shall maintain for a period of at least 2 years records of 6-minute average opacity levels for each separate control device.

(d) Each owner or operator subject to the provisions of § 61.186 shall maintain for a period of at least 2

years records of ambient inorganic arsenic concentrations at all sampling sites and other data needed to determine such concentrations.

(Approved by the Office of Management and Budget under control number 2060-0042)

§ 61.186 Reporting requirements.

(a) Each owner or operator subject to the provisions of § 61.183(a) shall provide the Administrator at least 30 days prior notice of each reference opacity level determination required in § 61.183(a) to afford the Administrator the opportunity to have an observer present.

(b) Each owner or operator subject to the provisions of § 61.183(a) shall submit to the Administrator:

(1) Within 60 days of conducting the evaluation required in § 61.183(b)(1), a written report of the continuous monitoring system evaluation;

(2) Within 30 days of establishing the reference opacity level required in § 61.183(d), a written report of the reference opacity level. The report shall also include the opacity data used and the calculations performed to determine the reference opacity level, and sufficient documentation to show that process and emission control equipment were operating normally during the reference opacity level determination; and

(3) A written report each quarter of each occurrence of excess opacity during the quarter. For the purposes of this paragraph, an occurrence of excess opacity is any 6-minute period during which the average opacity, as measured by the continuous monitoring system, exceeds the reference opacity level established under § 61.183(d).

(c) All quarterly reports of excess opacity shall be postmarked by the 30th day following the end of each quarter and shall include the following information:

(1) The magnitude of excess opacity, any conversion factor(s) used, and the dates and times of commencement and completion of each occurrence of excess opacity, the cause of each exceedance of the reference opacity

level, and the measures taken to minimize emissions.

(2) Specific identification of each period of excess opacity that occurred during startups, shutdowns, and malfunctions of the source.

(3) The date and time identifying each period during which the continuous monitoring system or monitoring device was inoperative, except for zero and span checks, and the nature of the system repairs or adjustments.

(d) Each owner or operator subject to this subpart shall submit a written report semiannually to the Administrator that describes the status and results, for the reporting period, of any pilot plant studies on alternative arsenic trioxide production processes. Conclusions and recommendations of the studies shall also be reported.

(e) All semiannual progress reports required in paragraph (d) of this section shall be postmarked by the 30th day following the end of each 6-month period.

(f) Each owner or operator of a source to which this subpart applies shall submit a written report each quarter to the Administrator that includes the following information:

(1) All ambient inorganic arsenic concentrations measured at all monitoring sites in accordance with § 61.184.

(2) A description of any modifications to the sampling network, during the reporting period, including any major maintenance, site changes, calibrations, and quality assurance information including sampling and analytical precision and accuracy estimates.

(g) All quarterly reports required in paragraph (f) of this section shall be postmarked by the 30th day following the end of each quarter.

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Subpart Q—National Emission Standards for Radon Emissions From Department of Energy Facilities

SOURCE: 54 FR 51701, Dec. 15, 1989, unless otherwise noted.

§ 61.190 Designation of facilities.

The provisions of this subpart apply to the design and operation of all storage and disposal facilities for radium-containing material (i.e., byproduct material as defined under section 11.e(2) of the Atomic Energy Act of 1954 (as amended)) that are owned or operated by the Department of Energy that emit radon-222 into air, including these facilities: The Feed Materials Production Center, Fernald, Ohio; the Niagara Falls Storage Site, Lewiston, New York; the Weldon Spring Site, Weldon Spring, Missouri; the Middlesex Sampling Plant, Middlesex, New Jersey; the Monticello Uranium Mill Tailings Pile, Monticello, Utah. This subpart does not apply to facilities listed in, or designated by the Secretary of Energy under title I of the Uranium Mill Tailings Control Act of 1978.

§ 61.191 Definitions.

As used in this subpart, all terms not defined here have the meaning given them in the Clean Air Act or subpart A of part 61. The following terms shall have the following specific meanings:

(a) *Facility* means all buildings, structures and operations on one contiguous site.

(b) *Source* means any building, structure, pile, impoundment or area used for interim storage or disposal that is or contains waste material containing radium in sufficient concentration to emit radon-222 in excess of this standard prior to remedial action.

§ 61.192 Standard.

No source at a Department of Energy facility shall emit more than 20 pCi/- m²-s of radon-222 as an average for the entire source, into the air. This requirement will be part of any Federal Facilities Agreement reached between Environmental Protection Agency and Department of Energy.

§ 61.193 Exemption from the reporting and testing requirements of 40 CFR 61.10.

All facilities designated under this subpart are exempt from the reporting requirements of 40 CFR 61.10.

Subpart R—National Emission Standards for Radon Emissions From Phosphogypsum Stacks

SOURCE: 54 FR 51701, Dec. 15, 1989, unless otherwise noted.

§ 61.200 Designation of facilities.

The provisions of this subpart apply to the owners and operators of the phosphogypsum that is produced as a result of phosphorus fertilizer production and all that is contained in existing phosphogypsum stacks.

§ 61.201 Definitions.

As used in this subpart, all terms not defined here have the meaning given them in the Clean Air Act or subpart A of part 61. The following terms shall have the following specific meanings:

(a) *Inactive stack* means a stack to which no further routine additions of phosphogypsum will be made and which is no longer used for water management associated with the production of phosphogypsum. If a stack has not been used for either purpose for two years it is presumed to be inactive.

(b) *Phosphogypsum stacks or stacks* are piles of waste from phosphorus fertilizer production containing phosphogypsum. Stacks shall also include phosphate mines that are used for the disposal of phosphogypsum.

§ 61.202 Standard.

All phosphogypsum shall be disposed of in stacks or in phosphate mines which shall not emit more than 20 pCi/m²-s of radon-222 into the air.

§ 61.203 Radon monitoring and compliance procedures.

(a) Sixty days following the date at which a stack becomes an inactive stack, or ninety days after the effective date of this rule if the stack is already inactive, the owners or operators of inactive phosphogypsum stacks shall test the stacks in accordance with the procedures described in 40 CFR part 61, Appendix B, Method 115. EPA shall be notified at least 30 days prior to an emissions test so that EPA may, at its option, observe the test. If meteorological conditions are such that a test cannot be properly conducted, then the owner or operator

shall notify EPA and test as soon as conditions permit.

(b) Ninety days after the testing is required, the owner or operator shall provide EPA with a report detailing the actions taken and the results of the radon-222 flux testing. Each report shall also include the following information:

(1) The name and location of the facility.

(2) A list of the stacks at the facility including the size and dimensions of the stack.

(3) The name of the person responsible for the operation of the facility and the name of the person preparing the report (if different).

(4) A description of the control measures taken to decrease the radon flux from the source and any actions taken to insure the long term effectiveness of the control measures, and

(5) The results of the testing conducted, including the results of each measurement.

(6) Each report shall be signed and dated by a corporate officer in charge of the facility and contain the following declaration immediately above the signature line: "I certify under penalty of law that I have personally examined and am familiar with the information submitted herein and based on my inquiry of those individuals immediately responsible for obtaining the information, I believe that the submitted information is true, accurate and complete. I am aware that there are significant penalties for submitting false information including the possibility of fine and imprisonment. See, 18 U.S.C. 1001."

(c) If year-long measurements are made in accordance with Method 115 Appendix B to part 61 this report shall include the results of the first measurement period and provide a schedule for the measurement frequency to be used. An additional report containing all the information in paragraph (b) of this section shall be submitted ninety days after completion of the final measurements.

(d) If at any point an owner or operator once again uses a stack for the disposal of phosphogypsum or for water management, the stack ceases to be in inactive status and the owner or

operator must notify EPA in writing within 45 days. When the owner or operator ceases to use the stack it will once again become inactive and require retesting and reporting.

(Approved by the Office of Management and Budget under control number 2060-0191)

§ 61.204 Recordkeeping requirements.

An owner or operator subject to this subpart must maintain records documenting the source of input parameters including the results of all measurements upon which they are based, the calculations and/or analytical methods used to derive values for input parameters, and the procedure used to determine compliance. This documentation should be sufficient to allow an independent auditor to verify the correctness of the determination made concerning the facility's compliance with the standard. These records must be kept by the owner or operator for at least five years and upon request be made available for inspection by the Administrator, or his authorized representative.

§ 61.205 Exemption from the reporting and testing requirements of 40 CFR 61.10.

All facilities designated under this subpart are exempt from the reporting requirements of 40 CFR 61.10.

Subpart S—[Reserved]

Subpart T—National Emission Standards for Radon Emissions From the Disposal of Uranium Mill Tailings

SOURCE: 54 FR 51702, Dec. 15, 1989, unless otherwise noted.

§ 61.220 Designation of facilities.

The provisions of this subpart apply to the owners and operators of all sites that are used for the disposal of tailings, and that managed residual radioactive material or uranium byproduct materials during and following the processing of uranium ores, commonly referred to as uranium mills and their associated tailings, that are listed in, or designated by the Secretary of

Energy under title I of the Uranium Mill Tailings Control Act of 1978 or regulated under title II of the Uranium Mill Tailings Control Act of 1978.

§ 61.221 Definitions.

As used in this subpart, all terms not defined here have the meaning given them in the Clean Air Act or subpart A of part 61. The following terms shall have the following specific meanings:

(a) *Long term stabilization* means the addition of material on a uranium mill tailings pile for purpose of ensuring compliance with the requirements of 40 CFR 192.02(a) or 192.32(b)(i). These actions shall be considered complete when the Nuclear Regulatory Commission determines that the requirements of 40 CFR 192.02(a) or 192.32(b)(i) have been met.

(b) *Operational* means a uranium mill tailings pile that is licensed to accept additional tailings, and those tailings can be added without violating subpart W or any other Federal, state or local rule or law. A pile cannot be considered operational if it is filled to capacity or the mill it accepts tailings from has been dismantled or otherwise decommissioned.

(c) *Uranium byproduct material or tailings* means the waste produced by the extraction or concentration of uranium from any ore processed primarily for its source material content. Ore bodies depleted by uranium solution extraction and which remain underground do not constitute byproduct material for the purposes of this subpart.

§ 61.222 Standard.

(a) Radon-222 emissions to the ambient air from uranium mill tailings pile that are no longer operational shall not exceed 20 pCi/m²-s of radon-222.

(b) Once a uranium mill tailings pile or impoundment ceases to be operational it must be disposed of and brought into compliance with this standard within two years of the effective date or within two years of the day it ceases to be operational whichever is later. If it is not physically possible for a mill owner or operator to complete disposal within that time, EPA shall, after consultation with the

mill owner or operator, establish a compliance agreement which will assure that disposal will be completed as quickly as possible.

§ 61.223 Compliance procedures.

(a) Sixty days following the completion of covering the pile to limit radon emissions but prior to the long term stabilization of the pile, the owners or operators of uranium mill tailings shall conduct testing for all piles within the facility in accordance with the procedures described in 40 CFR part 61, Appendix B, Method 115, or other procedures for which EPA has granted prior approval.

(b) Ninety days after the testing is required, each facility shall provide EPA with a report detailing the actions taken and the results of the radon-222 flux testing. EPA shall be notified at least 30 days prior to an emission test so that EPA may, at its option, observe the test. If meteorological conditions are such that a test cannot be properly conducted, then the owner or operator shall notify EPA and test as soon as conditions permit. Each report shall also include the following information:

(1) The name and location of the facility.

(2) A list of the piles at the facility.

(3) A description of the control measures taken to decrease the radon flux from the source and any actions taken to insure the long term effectiveness of the control measures.

(4) The results of the testing conducted, including the results of each measurement.

(5) Each report shall be signed and dated by a corporate officer or public official in charge of the facility and contain the following declaration immediately above the signature line: "I certify under penalty of law that I have personally examined and am familiar with the information submitted herein and based on my inquiry of those individuals immediately responsible for obtaining the information, I believe that the submitted information is true, accurate and complete. I am aware that there are significant penalties for submitting false information including the possibility of fine

and imprisonment. See, 18 U.S.C. 1001."

(c) If year long measurements are made in accordance with Method 115 of Appendix B of part 61, this report shall include the results of the first measurement period and provide a schedule for the measurement frequency to be used. An additional report shall be submitted ninety days after completion of the final measurements.

(d) If long term stabilization has begun before the effective date of the rule then testing may be conducted at any time, up to 60 days after the long term stabilization is completed.

(e) If the testing demonstrates that the pile meets the requirement of § 61.222(a) and long term stabilization has been completed then the pile is considered disposed for purposes of this rule.

(Approved by the Office of Management and Budget under control number 2060-0191)

§ 61.224 Recordkeeping requirements.

The owner or operator must maintain records documenting the source of input parameters including the results of all measurements upon which they are based, the calculations and/or analytical methods used to derive values for input parameters, and the procedure used to determine compliance. This documentation should be sufficient to allow an independent auditor to verify the accuracy of the determination made concerning the facility's compliance with the standard. The Administrator shall be kept apprised of the location of these records and the records must be kept for at least five years and upon request be made available for inspection by the Administrator, or his authorized representative.

§ 61.225 Exemption from the reporting and testing requirements of 40 CFR 61.10.

All facilities designated under this subpart are exempt from the reporting requirements of 40 CFR 61.10.

Subpart U—[Reserved]**Subpart V—National Emission Standard for Equipment Leaks (Fugitive Emission Sources)**

SOURCE: 49 FR 23513, June 6, 1984, unless otherwise noted.

§ 61.240 Applicability and designation of sources.

(a) The provisions of this subpart apply to each of the following sources that are intended to operate in volatile hazardous air pollutant (VHAP) service: pumps, compressors, pressure relief devices, sampling connection systems, open-ended valves or lines, valves, flanges and other connectors, product accumulator vessels, and control devices or systems required by this subpart.

(b) The provisions of this subpart apply to the sources listed in paragraph (a) after the date of promulgation of a specific subpart in part 61.

(c) While the provisions of this subpart are effective, a source to which this subpart applies that is also subject to the provisions of 40 CFR part 60 only will be required to comply with the provisions of this subpart.

§ 61.241 Definitions.

As used in this subpart, all terms not defined herein shall have the meaning given them in the Act, in subpart A of part 61, or in specific subparts of part 61; and the following terms shall have specific meaning given them:

Closed-vent system means a system that is not open to atmosphere and that is composed of piping, connections, and, if necessary, flow-inducing devices that transport gas or vapor from a piece or pieces of equipment to a control device.

Connector means flanged, screwed, welded, or other joined fittings used to connect two pipe lines or a pipe line and a piece of equipment. For the purpose of reporting and recordkeeping, connector means flanged fittings that are not covered by insulation or other materials that prevent location of the fittings.

Control device means an enclosed combustion device, vapor recovery system, or flare.

Double block and bleed system means two block valves connected in series with a bleed valve or line that can vent the line between the two block valves.

Equipment means each pump, compressor, pressure relief device, sampling connection system, open-ended valve or line, valve, flange or other connector, product accumulator vessel in VHAP service, and any control devices or systems required by this subpart.

First attempt at repair means to take rapid action for the purpose of stopping or reducing leakage of organic material to atmosphere using best practices.

In gas/vapor service means that a piece of equipment contains process fluid that is in the gaseous state at operating conditions.

In liquid service means that a piece of equipment is not in gas/vapor service.

In-situ sampling systems means non-extractive samplers or in-line samplers.

In vacuum service means that equipment is operating at an internal pressure which is at least 5 kilopascals (kPa) below ambient pressure.

In VHAP service means that a piece of equipment either contains or contacts a fluid (liquid or gas) that is at least 10 percent by weight a volatile hazardous air pollutant (VHAP) as determined according to the provisions of § 61.245(d). The provisions of § 61.245(d) also specify how to determine that a piece of equipment is not in VHAP service.

In VOC service means, for the purposes of this subpart, that (a) the piece of equipment contains or contacts a process fluid that is at least 10 percent VOC by weight (see 40 CFR 60.2 for the definition of volatile organic compound or VOC and 40 CFR 60.485(d) to determine whether a piece of equipment is not in VOC service) and (b) the piece of equipment is not in heavy liquid service as defined in 40 CFR 60.481.

Open-ended valve or line means any valve, except pressure relief valves, having one side of the valve seat in contact with process fluid and one side open to atmosphere, either directly or through open piping.

Pressure release means the emission of materials resulting from the system pressure being greater than the set pressure of the pressure relief device.

Process unit means equipment assembled to produce a VHAP or its derivatives as intermediates or final products, or equipment assembled to use a VHAP in the production of a product. A process unit can operate independently if supplied with sufficient feed or raw materials and sufficient product storage facilities.

Process unit shutdown means a work practice or operational procedure that stops production from a process unit or part of a process unit. An unscheduled work practice or operational procedure that stops production from a process unit or part of a process unit for less than 24 hours is not a process unit shutdown. The use of spare equipment and technically feasible bypassing of equipment without stopping production are not process unit shutdowns.

Product accumulator vessel means any distillate receiver, bottoms receiver, surge control vessel, or product separator in VHAP service that is vented to atmosphere either directly or through a vacuum-producing system. A product accumulator vessel is in VHAP service if the liquid or the vapor in the vessel is at least 10 percent by weight VHAP.

Repaired means that equipment is adjusted, or otherwise altered, to eliminate a leak.

Semiannual means a 6-month period; the first semiannual period concludes on the last day of the last month during the 180 days following initial startup for new sources; and the first semiannual period concludes on the last day of the last full month during the 180 days after the effective date of a specific subpart that references this subpart for existing sources.

Sensor means a device that measures a physical quantity or the change in a physical quantity, such as temperature, pressure, flow rate, pH, or liquid level.

Stuffing box pressure means the fluid (liquid or gas) pressure inside the casing or housing of a piece of equipment, on the process side of the in-board seal.

Volatile hazardous air pollutant or VHAP means a substance regulated under this part for which a standard for equipment leaks of the substance has been proposed and promulgated. Benzene is a VHAP. Vinyl chloride is a VHAP.

[49 FR 23513, June 6, 1984; 49 FR 38946, Oct. 2, 1984, as amended at 51 FR 34915, Sept. 30, 1986; 54 FR 38076, Sept. 14, 1989]

§ 61.242-1 Standards: General.

(a) Each owner or operator subject to the provisions of this subpart shall demonstrate compliance with the requirements of §§ 61.242-1 to 61.242-11 for each new and existing source as required in 40 CFR 61.05, except as provided in §§ 61.243 and 61.244.

(b) Compliance with this subpart will be determined by review of records, review of performance test results, and inspection using the methods and procedures specified in § 61.245.

(c)(1) An owner or operator may request a determination of alternative means of emission limitation to the requirements of §§ 61.242-2, 61.242-3, 61.242-5, 61.242-6, 61.242-7, 61.242-8, 61.242-9 and 61.242-11 as provided in § 61.244.

(2) If the Administrator makes a determination that a means of emission limitation is at least a permissible alternative to the requirements of §§ 61.242-2, 61.242-3, 61.242-5, 61.242-6, 61.242-7, 61.242-8, 61.242-9 or 61.242-11, an owner or operator shall comply with the requirements of that determination.

(d) Each piece of equipment to which this subpart applies shall be marked in such a manner that it can be distinguished readily from other pieces of equipment.

(e) Equipment that is in vacuum service is excluded from the requirements of § 61.242-2, to § 61.242-11 if it is identified as required in § 61.246(e)(5).

[49 FR 23513, June 6, 1984; 49 FR 38946, Oct. 2, 1984]

§ 61.242-2 Standards: Pumps.

(a)(1) Each pump shall be monitored monthly to detect leaks by the methods specified in § 61.245(b), except as

provided in § 61.242-1(c) and paragraphs (d), (e), and (f) of this section.

(2) Each pump shall be checked by visual inspection each calendar week for indications of liquids dripping from the pump seal.

(b)(1) If an instrument reading of 10,000 ppm or greater is measured, a leak is detected.

(2) If there are indications of liquids dripping from the pump seal, a leak is detected.

(c)(1) When a leak is detected, it shall be repaired as soon as practicable, but not later than 15 calendar days after it is detected, except as provided in § 61.242-10.

(2) A first attempt at repair shall be made no later than 5 calendar days after each leak is detected.

(d) Each pump equipped with a dual mechanical seal system that includes a barrier fluid system is exempt from the requirements of paragraph (a), provided the following requirements are met:

(1) Each dual mechanical seal system is:

(i) Operated with the barrier fluid at a pressure that is at all times greater than the pump stuffing box pressure; or

(ii) Equipped with a barrier fluid degassing reservoir that is connected by a closed-vent system to a control device that complies with the requirements of § 61.242-11; or

(iii) Equipped with a system that purges the barrier fluid into a process stream with zero VHAP emissions to atmosphere.

(2) The barrier fluid is not in VHAP service and, if the pump is covered by standards under 40 CFR part 60, is not in VOC service.

(3) Each barrier fluid system is equipped with a sensor that will detect failure of the seal system, the barrier fluid system, or both.

(4) Each pump is checked by visual inspection each calendar week for indications of liquids dripping from the pump seal.

(5)(i) Each sensor as described in paragraph (d)(3) of this section is checked daily or is equipped with an audible alarm, and

(ii) The owner or operator determines, based on design considerations

and operating experience, a criterion that indicates failure of the seal system, the barrier fluid system, or both.

(6)(i) If there are indications of liquids dripping from the pump seal or the sensor indicates failure of the seal system, the barrier fluid system, or both based on the criterion determined in paragraph (d)(5)(ii), a leak is detected.

(ii) When a leak is detected, it shall be repaired as soon as practicable, but not later than 15 calendar days after it is detected, except as provided in § 61.242-10.

(iii) A first attempt at repair shall be made no later than 5 calendar days after each leak is detected.

(e) Any pump that is designated, as described in § 61.246(e)(2), for no detectable emissions, as indicated by an instrument reading of less than 500 ppm above background, is exempt from the requirements of paragraphs (a), (c), and (d) if the pump:

(1) Has no externally actuated shaft penetrating the pump housing,

(2) Is demonstrated to be operating with no detectable emissions, as indicated by an instrument reading of less than 500 ppm above background, as measured by the method specified in § 61.245(c), and

(3) Is tested for compliance with paragraph (e)(2) initially upon designation, annually, and at other times requested by the Administrator.

(f) If any pump is equipped with a closed-vent system capable of capturing and transporting any leakage from the seal or seals to a control device that complies with the requirements of § 61.242-11, it is exempt from the requirements of paragraphs (a)-(e).

(g) Any pump that is located within the boundary of an unmanned plant site is exempt from the weekly visual inspection requirement of paragraphs (a)(2) and (d)(4) of this section, and the daily requirements of paragraph (d)(5)(i) of this section, provided that each pump is visually inspected as often as practicable and at least monthly.

[49 FR 23513, June 6, 1984, as amended at 49 FR 38946, Oct. 2, 1984]

§ 61.242-3 Standards: Compressors.

(a) Each compressor shall be equipped with a seal system that includes a barrier fluid system and that prevents leakage of process fluid to atmosphere, except as provided in § 61.242-1(c) and paragraphs (h) and (i) of this section.

(b) Each compressor seal system as required in paragraph (a) shall be:

(1) Operated with the barrier fluid at a pressure that is greater than the compressor stuffing box pressure; or

(2) Equipped with a barrier fluid system that is connected by a closed-vent system to a control device that complies with the requirements of § 61.242-11; or

(3) Equipped with a system that purges the barrier fluid into a process stream with zero VHAP emissions to atmosphere.

(c) The barrier fluid shall not be in VHAP service and, if the compressor is covered by standards under 40 CFR part 60, shall not be in VOC service.

(d) Each barrier fluid system as described in paragraphs (a)-(c) of this section shall be equipped with a sensor that will detect failure of the seal system, barrier fluid system, or both.

(e)(1) Each sensor as required in paragraph (d) of this section shall be checked daily or shall be equipped with an audible alarm unless the compressor is located within the boundary of an unmanned plant site.

(2) The owner or operator shall determine, based on design considerations and operating experience, a criterion that indicates failure of the seal system, the barrier fluid system, or both.

(f) If the sensor indicates failure of the seal system, the barrier fluid system, or both based on the criterion determined under paragraph (e)(2) of this section, a leak is detected.

(g)(1) When a leak is detected, it shall be repaired as soon as practicable, but not later than 15 calendar days after it is detected, except as provided in § 61.242-10.

(2) A first attempt at repair shall be made no later than 5 calendar days after each leak is detected.

(h) A compressor is exempt from the requirements of paragraphs (a) and (b) if it is equipped with a closed-vent

system capable of capturing and transporting any leakage from the seal to a control device that complies with the requirements of § 61.242-11, except as provided in paragraph (i).

(i) Any Compressor that is designated, as described in § 61.246(e)(2), for no detectable emission as indicated by an instrument reading of less than 500 ppm above background is exempt from the requirements of paragraphs (a)-(h) if the compressor:

(1) Is demonstrated to be operating with no detectable emissions, as indicated by an instrument reading of less than 500 ppm above background, as measured by the method specified in § 61.245(c); and

(2) Is tested for compliance with paragraph (i)(1) initially upon designation, annually, and at other times requested by the Administrator.

[49 FR 23513, June 6, 1984; 49 FR 38946, Oct. 2, 1984]

§ 61.242-4 Standards: Pressure relief devices in gas/vapor service.

(a) Except during pressure releases, each pressure relief device in gas/vapor service shall be operated with no detectable emissions, as indicated by an instrument reading of less than 500 ppm above background, as measured by the method specified in § 61.245(c).

(b)(1) After each pressure release, the pressure relief device shall be returned to a condition of no detectable emissions, as indicated by an instrument reading of less than 500 ppm above background, as soon as practicable, but no later than 5 calendar days after each pressure release, except as provided in § 61.242-10.

(2) No later than 5 calendar days after the pressure release, the pressure relief device shall be monitored to confirm the condition of no detectable emissions, as indicated by an instrument reading of less than 500 ppm above background, as measured by the method specified in § 61.245(c).

(c) Any pressure relief device that is equipped with a closed-vent system capable of capturing and transporting leakage from the pressure relief device to a control device as described in

§ 61.242-11 is exempt from the requirements of paragraphs (a) and (b).

[49 FR 23513, June 6, 1984; 49 FR 38946, Oct. 2, 1984]

§ 61.242-5 Standards: Sampling connecting systems.

(a) Each sampling connection system shall be equipped with a closed-purge system or closed vent system, except as provided in § 61.242-1(c).

(b) Each closed-purge system or closed-vent system as required in paragraph (a) shall:

(1) Return the purged process fluid directly to the process line with zero VHAP emissions to atmosphere; or

(2) Collect and recycle the purged process fluid with zero VHAP emissions to atmosphere; or

(3) Be designed and operated to capture and transport all the purged process fluid to a control device that complies with the requirements of § 61.242-11.

(c) *In-situ* sampling systems are exempt from the requirements of paragraphs (a) and (b).

§ 61.242-6 Standards: Open-ended valves or lines.

(a)(1) Each open-ended valve or line shall be equipped with a cap, blind flange, plug, or a second valve, except as provided in § 61.242-1(c).

(2) The cap, blind flange, plug, or second valve shall seal the open end at all times except during operations requiring process fluid flow through the open-ended valve or line.

(b) Each open-ended valve or line equipped with a second valve shall be operated in a manner such that the valve on the process fluid end is closed before the second valve is closed.

(c) When a double block and bleed system is being used, the bleed valve or line may remain open during operations that require venting the line between the block valves but shall comply with paragraph (a) at all other times.

§ 61.242-7 Standards: Valves.

(a) Each valve shall be monitored monthly to detect leaks by the method specified in § 61.245(b) and shall comply with paragraphs (b)-(e), except as provided in paragraphs (f),

(g), and (h) of this section, § 61.243-1 or § 61.243-2, and § 61.242-1(c).

(b) If an instrument reading of 10,000 ppm or greater is measured, a leak is detected.

(c)(1) Any valve for which a leak is not detected for 2 successive months may be monitored the first month of every quarter, beginning with the next quarter, until a leak is detected.

(2) If a leak is detected, the valve shall be monitored monthly until a leak is not detected for 2 successive months.

(d)(1) When a leak is detected, it shall be repaired as soon as practicable, but no later than 15 calendar days after the leak is detected, except as provided in § 61.242-10.

(2) A first attempt at repair shall be made no later than 5 calendar days after each leak is detected.

(e) First attempts at repair include, but are not limited to, the following best practices where practicable:

(1) Tightening of bonnet bolts;
(2) Replacement of bonnet bolts;
(3) Tightening of packing gland nuts; and

(4) Injection of lubricant into lubricated packing.

(f) Any valve that is designated, as described in § 61.246(e)(2), for no detectable emissions, as indicated by an instrument reading of less than 500 ppm above background, is exempt from the requirements of paragraph (a) if the valve:

(1) Has no external actuating mechanism in contact with the process fluid;

(2) Is operated with emissions less than 500 ppm above background, as measured by the method specified in § 61.245(c); and

(3) Is tested for compliance with paragraph (f)(2) initially upon designation, annually, and at other times requested by the Administrator.

(g) Any valve that is designated, as described in § 61.246(f)(1), as an unsafe-to-monitor valve is exempt from the requirements of paragraph (a) if:

(1) The owner or operator of the valve demonstrates that the valve is unsafe to monitor because monitoring personnel would be exposed to an im-

mediate danger as a consequence of complying with paragraph (a); and

(2) The owner or operator of the valve has a written plan that requires monitoring of the valve as frequent as practicable during safe-to-monitor times.

(h) Any valve that is designated, as described in § 61.246(f)(2), as a difficult-to-monitor valve is exempt from the requirements of paragraph (a) if:

(1) The owner or operator of the valve demonstrates that the valve cannot be monitored without elevating the monitoring personnel more than 2 meters above a support surface;

(2) The process unit within which the valve is located is an existing process unit; and

(3) The owner or operator of the valve follows a written plan that requires monitoring of the valve at least once per calendar year.

§ 61.242-8 Standards: Pressure relief devices in liquid service and flanges and other connectors.

(a) Pressure relief devices in liquid service and flanges and other connectors shall be monitored within 5 days by the method specified in § 61.245(b) if evidence of a potential leak is found by visual, audible, olfactory, or any other detection method, except as provided in § 61.242-1(c).

(b) If an instrument reading of 10,000 ppm or greater is measured, a leak is detected.

(c)(1) When a leak is detected, it shall be repaired as soon as practicable, but not later than 15 calendar days after it is detected, except as provided in § 61.242-10.

(2) The first attempt at repair shall be made no later than 5 calendar days after each leak is detected.

(d) First attempts at repair include, but are not limited to, the best practices described under § 61.242-7(e).

[49 FR 23513, June 6, 1984; 49 FR 38946, Oct. 2, 1984]

§ 61.242-9 Standards: Product accumulator vessels.

Each product accumulator vessel shall be equipped with a closed-vent system capable of capturing and transporting any leakage from the vessel to a control device as described in

§ 61.242-11, except as provided in § 61.242-1(c).

[49 FR 23513, June 6, 1984; 49 FR 38946, Oct. 2, 1984]

§ 61.242-10 Standards: Delay of repair.

(a) Delay of repair of equipment for which leaks have been detected will be allowed if the repair is technically infeasible without a process unit shutdown. Repair of this equipment shall occur before the end of the next process unit shutdown.

(b) Delay of repair of equipment for which leaks have been detected will be allowed for equipment that is isolated from the process and that does not remain in VHAP service.

(c) Delay of repair for valves will be allowed if:

(1) The owner or operator demonstrates that emissions of purged material resulting from immediate repair are greater than the fugitive emissions likely to result from delay of repair, and

(2) When repair procedures are effected, the purged material is collected and destroyed or recovered in a control device complying with § 61.242-11.

(d) Delay of repair for pumps will be allowed if:

(1) Repair requires the use of a dual mechanical seal system that includes a barrier fluid system, and

(2) Repair is completed as soon as practicable, but not later than 6 months after the leak was detected.

(e) Delay of repair beyond a process unit shutdown will be allowed for a valve if valve assembly replacement is necessary during the process unit shutdown, valve assembly supplies have been depleted, and valve assembly supplies had been sufficiently stocked before the supplies were depleted. Delay of repair beyond the next process unit shutdown will not be allowed unless the next process unit shutdown occurs sooner than 6 months after the first process unit shutdown.

§ 61.242-11 Standards: Closed-vent systems and control devices.

(a) Owners or operators of closed-vent systems and control devices used to comply with provisions of this sub-

part shall comply with the provisions of this section, except as provided in § 61.242-1(c).

(b) Vapor recovery systems (for example, condensers and adsorbers) shall be designed and operated to recover the organic vapors vented to them with an efficiency of 95 percent or greater.

(c) Enclosed combustion devices shall be designed and operated to reduce the VHAP emissions vented to them with an efficiency of 95 percent or greater or to provide a minimum residence time of 0.50 seconds at a minimum temperature of 760°C.

(d) Flares shall be used to comply with this subpart shall comply with the requirements of § 60.18.

(e) Owners or operators of control devices that are used to comply with the provisions of this subpart shall monitor these control devices to ensure that they are operated and maintained in conformance with their design.

(f)(1) Closed-vent systems shall be designed for and operated with no detectable emissions, as indicated by an instrument reading of less than 500 ppm above background and by visual inspections, as determined by the methods specified in § 61.245(c).

(2) Closed-event systems shall be monitored to determine compliance with this section initially in accordance with § 61.05, annually, and at other times requested by the administrator.

(3) Leaks, as indicated by an instrument reading greater than 500 ppm and visual inspections, shall be repaired as soon as practicable, but not later than 15 calendar days after the leak is detected.

(4) A first attempt at repair shall be made no later than 5 calendar days after the leak is detected.

(g) Closed-vent systems and control devices use to comply with provisions of this subpart shall be operated at all times when emissions may be vented to them.

[49 FR 23513, June 6, 1984; 49 FR 38946, Oct. 2, 1984, as amended at 51 FR 2702, Jan. 21, 1986]

§ 61.243-1 Alternative standards for valves in VHAP service—allowable percentage of valves leaking.

(a) An owner or operator may elect to have all valves within a process unit to comply with an allowable percentage of valves leaking of equal to or less than 2.0 percent.

(b) The following requirements shall be met if an owner or operator decides to comply with an allowable percentage of valves leaking:

(1) An owner or operator must notify the Administrator that the owner or operator has elected to have all valves within a process unit to comply with the allowable percentage of valves leaking before implementing this alternative standard, as specified in § 61.247(d).

(2) A performance test as specified in paragraph (c) of this section shall be conducted initially upon designation, annually, and at other times requested by the Administrator.

(3) If a valve leak is detected, it shall be repaired in accordance with § 61.242-7(d) and (e).

(c) Performance tests shall be conducted in the following manner:

(1) All valves in VHAP service within the process unit shall be monitored within 1 week by the methods specified in § 61.245(b).

(2) If an instrument reading of 10,000 ppm or greater is measured, a leak is detected.

(3) The leak percentage shall be determined by dividing the number of valves in VHAP service for which leaks are detected by the number of valves in VHAP service within the process unit.

(d) Owner or operators who elect to have all valves comply with this alternative standard shall not have a process unit with a leak percentage greater than 2.0 percent.

(e) If an owner or operator decides no longer to comply with § 61.243-1, the owner or operator must notify the Administrator in writing that the work practice standard described in § 61.242-7(a)-(e) will be followed.

§ 61.243-2 Alternative standards for valves in VHAP service—skip period leak detection and repair.

(a)(1) An owner or operator may elect for all valves within a process unit to comply with one of the alternative work practices specified in paragraphs (b)(2) and (3) of this section.

(2) An owner or operator must notify the Administrator before implementing one of the alternative work practices, as specified in § 61.247(d).

(b)(1) An owner or operator shall comply initially with the requirements for valves, as described in § 61.242-7.

(2) After 2 consecutive quarterly leak detection periods with the percentage of valves leaking equal to or less than 2.0, an owner or operator may begin to skip 1 of the quarterly leak detection periods for the valves in VHAP service.

(3) After 5 consecutive quarterly leak detection periods with the percentage of valves leaking equal to or less than 2.0, an owner or operator may begin to skip 3 of the quarterly leak detection periods for the valves in VHAP service.

(4) If the percentage of valves leaking is greater than 2.0, the owner or operator shall comply with the requirements as described in § 61.242-7 but may again elect to use this section.

§ 61.244 Alternative means of emission limitation.

(a) Permission to use an alternative means of emission limitation under section 112(e)(3) of the Clean Air Act shall be governed by the following procedures:

(b) Where the standard is an equipment, design, or operational requirement:

(1) Each owner or operator applying for permission shall be responsible for collecting and verifying test data for an alternative means of emission limitation. Limitation to test data for the equipment, design, and operational requirements.

(3) The Administrator may condition the permission on requirements that may be necessary to assure operation and maintenance to achieve the same emission reduction as the equipment, design, and operational requirements.

(c) Where the standard is a work practice:

(1) Each owner or operator applying for permission shall be responsible for collecting and verifying test data for an alternative means of emission limitation.

(2) For each source for which permission is requested, the emission reduction achieved by the required work practices shall be demonstrated for a minimum period of 12 months.

(3) For each source for which permission is requested, the emission reduction achieved by the alternative means of emission limitation shall be demonstrated.

(4) Each owner or operator applying for permission shall commit in writing each source to work practices that provide for emission reductions equal to or greater than the emission reductions achieved by the required work practices.

(5) The Administrator will compare the demonstrated emission reduction for the alternative means of emission limitation to the demonstrated emission reduction for the required work practices and will consider the commitment in paragraph (c)(4).

(6) The Administrator may condition the permission on requirements that may be necessary to assure operation and maintenance to achieve the same emission reduction as the required work practices of this subpart.

(d) An owner or operator may offer a unique approach to demonstrate the alternative means of emission limitation.

(e)(1) Manufacturers of equipment used to control equipment leaks of a VHAP may apply to the Administrator for permission for an alternative means of emission limitation that achieves a reduction in emissions of the VHAP achieved by the equipment, design, and operational requirements of this subpart.

(2) The Administrator will grant permission according to the provisions of paragraphs (b), (c), and (d).

§ 61.245 Test methods and procedures.

(a) Each owner or operator subject to the provisions of this subpart shall comply with the test methods and pro-

cedures requirements provided in this section.

(b) Monitoring, as required in §§ 61.242, 61.243, 61.244, and 61.135, shall comply with the following requirements:

(1) Monitoring shall comply with Method 21 of Appendix A of 40 CFR part 60.

(2) The detection instrument shall meet the performance criteria of Reference Method 21.

(3) The instrument shall be calibrated before use on each day of its use by the procedures specified in Reference Method 21.

(4) Calibration gases shall be:

(i) Zero air (less than 10 ppm of hydrocarbon in air); and

(ii) A mixture of methane or n-hexane and air at a concentration of approximately, but less than, 10,000 ppm methane or n-hexane.

(5) The instrument probe shall be traversed around all potential leak interfaces as close to the interface as possible as described in Reference Method 21.

(c) When equipment is tested for compliance with or monitored for no detectable emissions, the owner or operator shall comply with the following requirements:

(1) The requirements of paragraphs (b) (1) through (4) shall apply.

(2) The background level shall be determined, as set forth in Reference Method 21.

(3) The instrument probe shall be traversed around all potential leak interfaces as close to the interface as possible as described in Reference Method 21.

(4) The arithmetic difference between the maximum concentration indicated by the instrument and the background level is compared with 500 ppm for determining compliance.

(d)(1) Each piece of equipment within a process unit that can conceivably contain equipment in VHAP service is presumed to be in VHAP service unless an owner or operator demonstrates that the piece of equipment is not in VHAP service. For a piece of equipment to be considered not in VHAP service, it must be determined that the percent VHAP content can be reasonably expected never to exceed

10 percent by weight. For purposes of determining the percent VHAP content of the process fluid that is contained in or contacts equipment, procedures that conform to the methods described in ASTM Method D-2267 (incorporated by the reference as specified in § 61.18) shall be used.

(2)(i) An owner or operator may use engineering judgment rather than the procedures in paragraph (d)(1) of this section to demonstrate that the percent VHAP content does not exceed 10 percent by weight, provided that the engineering judgment demonstrates that the VHAP content clearly does not exceed 10 percent by weight. When an owner or operator and the Administrator do not agree on whether a piece of equipment is not in VHAP service, however, the procedures in paragraph (d)(1) of this section shall be used to resolve the disagreement.

(ii) If an owner or operator determines that a piece of equipment is in VHAP service, the determination can be revised only after following the procedures in paragraph (d)(1) of this section.

(3) Samples used in determining the percent VHAP content shall be representative of the process fluid that is contained in or contacts the equipment or the gas being combusted in the flare.

(e)(1) Method 22 of Appendix A of 40 CFR part 60 shall be used to determine compliance of flares with the visible emission provisions of this subpart.

(2) The presence of a flare pilot flame shall be monitored using a thermocouple or any other equivalent device to detect the presence of a flame.

(3) The net heating value of the gas being combusted in a flare shall be calculated using the following equation:

$$H_T = K \left(\sum_{i=1}^n C_i H_i \right)$$

Where:

H_T = Net heating value of the sample, MJ/scm; where the net enthalpy per mole of offgas is based on combustion at 25°C and 760 mm Hg, but the standard temperature for determining the volume corresponding to one mole is 20°C.

K = Constant, 1.74×10^{-7} (1/ppm) (g mole/scm) (MJ/kcal) where standard temperature for (g mole/scm) is 20°C

C_i = Concentration of sample component i in ppm, as measured by Reference Method 18 of Appendix A of 40 FR part 60 and ASTM D2504-67 (reapproved 1977) (incorporated by reference as specified in § 61.18).

H_i = Net heat of combustion of sample component i , kcal/g mole. The heats of combustion may be determined using ASTM D2382-76 (incorporated by reference as specified in § 61.18) if published values are not available or cannot be calculated.

(4) The actual exit velocity of a flare shall be determined by dividing the volumetric flowrate (in units of standard temperature and pressure), as determined by Reference Method 2, 2A, 2C, or 2D, as appropriate, by the unobstructed (free) cross section area of the flare tip.

(5) The maximum permitted velocity, V_{max} , for air-assisted flares shall be determined by the following equation:

$$V_{Max} = 8.76 + 0.7084(H_T)$$

Where:

V_{Max} = Maximum permitted velocity, m/sec

8.706 = Constant.

0.7084 = Constant.

H_T = The net heating value as determined in paragraph (e)(3) of this section.

[49 FR 23513, June 6, 1984, as amended at 49 FR 38946, Oct. 2, 1984; 49 FR 43647, Oct. 31, 1984; 53 FR 36972, Sept. 23, 1988; 54 FR 38077, Sept. 14, 1989]

§ 61.246 Recordkeeping requirements.

(a)(1) Each owner or operator subject to the provisions of this subpart shall comply with the recordkeeping requirements of this section.

(2) An owner or operator of more than one process unit subject to the provisions of this subpart may comply with the recordkeeping requirements for these process units in one recordkeeping system if the system identifies each record by each process unit.

(b) When each leak is detected as specified in §§ 61.242-2, 61.242-3, 61.242-7, 61.242-8, and 61.135, the following requirements apply:

(1) A weatherproof and readily visible identification, marked with the equipment identification number, shall be attached to the leaking equipment.

(2) The identification on a valve may be removed after it has been monitored for 2 successive months as specified in § 61.242-7(c) and no leak has been detected during those 2 months.

(3) The identification on equipment, except on a valve, may be removed after it has been repaired.

(c) When each leak is detected as specified in 61.242-2, 61.242-3, 61.242-7, 61.242-8, and 61.135, the following information shall be recorded in a log and shall be kept for 2 years in a readily accessible location:

(1) The instrument and operator identification numbers and the equipment identification number.

(2) The date the leak was detected and the dates of each attempt to repair the leak.

(3) Repair methods applied in each attempt to repair the leak.

(4) "Above 10,000" if the maximum instrument reading measured by the methods specified in § 61.245(a) after each repair attempt is equal to or greater than 10,000 ppm.

(5) "Repair delayed" and the reason for the delay if a leak is not repaired within 15 calendar days after discovery of the leak.

(6) The signature of the owner or operator (or designate) whose decision it was that repair could not be effected without a process shutdown.

(7) The expected date of successful repair of the leak if a leak is not repaired within 15 calendar days.unrepaired.

(9) The date of successful repair of the leak.

(d) The following information pertaining to the design requirements for closed-vent systems and control devices described in § 61.242-11 shall be recorded and kept in a readily accessible location:

(1) Detailed schematics, design specifications, and piping and instrumentation diagrams.

(2) The dates and descriptions of any changes in the design specifications.

(3) A description of the parameter or parameters monitored, as required in § 61.242-11(e), to ensure that control devices are operated and maintained in conformance with their design and an explanation of why that parameter (or parameters) was selected for the monitoring.

(4) Periods when the closed-vent systems and control devices required in §§ 61.242-2, 61.242-3, 61.242-4, 61.242-5 and 61.242-9 are not operated as designed, including periods when a flare pilot light does not have a flame.

(5) Dates of startups and shutdowns of the closed-vent systems and control devices required in §§ 61.242-2, 61.242-3, 61.242-4, 61.242-5 and 61.242-9.

(e) The following information pertaining to all equipment to which a standard applies shall be recorded in a log that is kept in a readily accessible location:

(1) A list of identification numbers for equipment (except welded fittings) subject to the requirements of this subpart.

(2)(i) A list of identification numbers for equipment that the owner or operator elects to designate for no detectable emissions as indicated by an instrument reading of less than 500 ppm above background.

(ii) The designation of this equipment for no detectable emissions shall be signed by the owner or operator.

(3) A list of equipment identification numbers for pressure relief devices required to comply with § 61.242-4(a).

(4)(i) The dates of each compliance test required in §§ 61.242-2(e), 61.242-3(i), 61.242-4, 61.242-7(f), and 61.135(g).

(ii) The background level measured during each compliance test.

(iii) The maximum instrument reading measured at the equipment during each compliance test.

(5) A list of identification numbers for equipment in vacuum service.

(f) The following information pertaining to all valves subject to the requirements of § 61.242-7(g) and (h) shall be recorded in a log that is kept in a readily accessible location:

(1) A list of identification numbers for valves that are designated as unsafe to monitor, an explanation for each valve stating why the valve is

unsafe to monitor, and the plan for monitoring each valve.

(2) A list of identification numbers for valves that are designated as difficult to monitor, an explanation for each valve stating why the valve is difficult to monitor, and the planned schedule for monitoring each valve.

(g) The following information shall be recorded for valves complying with § 61.243-2:

(1) A schedule of monitoring.

(2) The percent of valves found leaking during each monitoring period.

(h) The following information shall be recorded in a log that is kept in a readily accessible location:

(1) Design criterion required in §§ 61.242-2(d)(5), 61.242-3(e)(2), and 61.135(e)(4) and an explanation of the design criterion; and

(2) Any changes to this criterion and the reasons for the changes.

(i) The following information shall be recorded in a log that is kept in a readily accessible location for use in determining exemptions as provided in the applicability section of this subpart and other specific subparts:

(1) An analysis demonstrating the design capacity of the process unit, and

(2) An analysis demonstrating that equipment is not in VHAP service.

(j) Information and data used to demonstrate that a piece of equipment is not in VHAP service shall be recorded in a log that is kept in a readily accessible location.

(Approved by the Office of Management and Budget under control number 2060-0068)

[49 FR 23513, June 6, 1984, as amended at 49 FR 38946, Oct. 2, 1984; 54 FR 38077, Sept. 14, 1989]

§ 61.247 Reporting requirements.

(a)(1) An owner or operator of any piece of equipment to which this subpart applies shall submit a statement in writing notifying the Administrator that the requirements of §§ 61.242, 61.245, 61.246, and 61.247 are being implemented.

(2) In the case of an existing source or a new source which has an initial startup date preceding the effective date, the statement is to be submitted

within 90 days of the effective date, unless a waiver of compliance is granted under § 61.11, along with the information required under § 61.10. If a waiver of compliance is granted, the statement is to be submitted on a date scheduled by the Administrator.

(3) In the case of new sources which did not have an initial startup date preceding the effective date, the statement shall be submitted with the application for approval of construction, as described in § 61.07.

(4) The statement is to contain the following information for each source:

(i) Equipment identification number and process unit identification.

(ii) Type of equipment (for example, a pump or pipeline valve).

(iii) Percent by weight VHAP in the fluid at the equipment.

(iv) Process fluid state at the equipment (gas/vapor or liquid).

(v) Method of compliance with the standard (for example, "monthly leak detection and repair" or "equipped with dual mechanical seals").

(b) A report shall be submitted to the Administrator semiannually starting 6 months after the initial report required in paragraph (a) of this section, that includes the following information:

(1) Process unit identification.

(2) For each month during the semiannual reporting period,

(i) Number of valves for which leaks were detected as described in § 61.242-7(b) of § 61.243-2.

(ii) Number of valves for which leaks were not repaired as required in § 61.242-7(d).

(iii) Number of pumps for which leaks were detected as described in § 61.242-2 (b) and (d)(6).

(iv) Number of pumps for which leaks were not repaired as required in § 61.242-2 (c) and (d)(6).

(v) Number of compressors for which leaks were detected as described in § 61.242-3(f).

(vi) Number of compressors for which leaks were not repaired as required in § 61.242-3(g).

(vii) The facts that explain any delay of repairs and, where appropriate, why a process unit shutdown was technically infeasible.

(3) Dates of process unit shutdowns which occurred within the semiannual reporting period.

(4) Revisions to items reported according to paragraph (a) if changes have occurred since the initial report or subsequent revisions to the initial report.

NOTE: Compliance with the requirements of § 61.10(c) is not required for revisions documented under this paragraph.

(5) The results of all performance tests and monitoring to determine compliance with no detectable emissions and with §§ 61.243-1 and 61.243-2 conducted within the semiannual reporting period.

(c) In the first report submitted as required in paragraph (a) of this section, the report shall include a reporting schedule stating the months that semiannual reports shall be submitted. Subsequent reports shall be submitted according to that schedule, unless a revised schedule has been submitted in a previous semiannual report.

(d) An owner or operator electing to comply with the provisions of §§ 61.243-1 and 61.243-2 shall notify the Administrator of the alternative standard selected 90 days before implementing either of the provisions.

(e) An application for approval of construction or modification, §§ 61.05(a) and 61.07, will not be required if—

(1) The new source complies with the standard, § 61.242;

(2) The new source is not part of the construction of a process unit; and

(3) In the next semiannual report required by paragraph (b) of this section, the information in paragraph (a)(4) of this section is reported.

(Approved by the Office of Management and Budget under control number ICR-1153)

[49 FR 23513, June 6, 1984, as amended at 49 FR 38947, Oct. 2, 1984; 54 FR 38077, Sept. 14, 1989]

Subpart W—National Emission Standards for Radon Emissions From Operating Mill Tailings

SOURCE: 54 FR 51703, Dec. 15, 1989, unless otherwise noted.

§ 61.250

§ 61.250 Designation of facilities.

The provisions of this subpart apply to owners or operators of facilities licensed to manage uranium byproduct materials during and following the processing of uranium ores, commonly referred to as uranium mills and their associated tailings. This subpart does not apply to the disposal of tailings.

§ 61.251 Definitions.

As used in this subpart, all terms not defined here have the meaning given them in the Clean Air Act or 40 CFR part 61, subpart A. The following terms shall have the following specific meanings:

(a) *Area* means the vertical projection of the pile upon the earth's surface.

(b) *Continuous disposal* means a method of tailings management and disposal in which tailings are dewatered by mechanical methods immediately after generation. The dried tailings are then placed in trenches or other disposal areas and immediately covered to limit emissions consistent with applicable Federal standards.

(c) *Dewatered* means to remove the water from recently produced tailings by mechanical or evaporative methods such that the water content of the tailings does not exceed 30 percent by weight.

(d) *Existing impoundment* means any uranium mill tailings impoundment which is licensed to accept additional tailings and is in existence as of December 15, 1989.

(e) *Operation* means that an impoundment is being used for the continued placement of new tailings or is in standby status for such placement. An impoundment is in operation from the day that tailings are first placed in the impoundment until the day that final closure begins.

(f) *Phased disposal* means a method of tailings management and disposal which uses lined impoundments which are filled and then immediately dried and covered to meet all applicable Federal standards.

(g) *Uranium byproduct material or tailings* means the waste produced by the extraction or concentration of uranium from any ore processed primarily for its source material content. Ore

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bodies depleted by uranium solution extraction and which remain underground do not constitute byproduct material for the purposes of this subpart.

§ 61.252 Standard.

(a) Radon-222 emissions to the ambient air from an existing uranium mill tailings pile shall not exceed 20 pCi/m²-s of radon-222.

(b) After December 15, 1989, no new tailings impoundment can be built unless it is designed, constructed and operated to meet one of the two following work practices:

(1) Phased disposal in lined tailings impoundments that are no more than 40 acres in area and meet the requirements of 40 CFR 192.32(a) as determined by the Nuclear Regulatory Commission. The owner or operator shall have no more than two impoundments, including existing impoundments, in operation at any one time.

(2) Continuous disposal of tailings such that tailings are dewatered and immediately disposed with no more than 10 acres uncovered at any time and operated in accordance with § 192.32(a) as determined by the Nuclear Regulatory Commission.

(c) All mill owners or operators shall comply with the provisions of 40 CFR 192.32(a) in the operation of tailings piles, the exemption for existing piles in 40 CFR 192.32(a) notwithstanding.

§ 61.253 Determining compliance.

Compliance with the emission standard in this subpart shall be determined annually through the use of Method 115 of Appendix B. When measurements are to be made over a one year period, EPA shall be provided with a schedule of the measurement frequency to be used. The schedule may be submitted to EPA prior to or after the first measurement period. EPA shall be notified 30 days prior to any emissions test so that EPA may, at its option, observe the test.

§ 61.254 Annual reporting requirements.

(a) The owners or operators of operating existing mill impoundments shall report the results of the compliance calculations required in § 61.253

and the input parameters used in making the calculation for each calendar year shall be sent to EPA by March 31 of the following year. Each report shall also include the following information:

(1) The name and location of the mill.

(2) The name of the person responsible for the operation of the facility and the name of the person preparing the report (if different).

(3) The results of the testing conducted, including the results of each measurement.

(4) Each report shall be signed and dated by a corporate officer in charge of the facility and contain the following declaration immediately above the signature line: "I certify under penalty of law that I have personally examined and am familiar with the information submitted herein and based on my inquiry of those individuals immediately responsible for obtaining the information, I believe that the submitted information is true, accurate and complete. I am aware that there are significant penalties for submitting false information including the possibility of fine and imprisonment. See, 18 U.S.C. 1001."

(b) If the facility is not in compliance with the emission limits of § 61.252 in the calendar year covered by the report, then the facility must commence reporting to the Administrator on a monthly basis the information listed in paragraph (a) of this section, for the preceding month. These reports will start the month immediately following the submittal of the annual report for the year in noncompliance and will be due 30 days following the end of each month. This increased level of reporting will continue until the Administrator has determined that the monthly reports are no longer necessary. In addition to all the information required in paragraph (a) of this section, monthly reports shall also include the following information:

(1) All controls or other changes in operation of the facility that will be or are being installed to bring the facility into compliance.

(2) If the facility is under a judicial or administrative enforcement decree, the report will describe the facilities

performance under the terms of the decree.

(c) The first report will cover the emissions of calendar year 1990.

(Approved by the Office of Management and Budget under control number 2060-0191)

§ 61.255 Recordkeeping requirements.

The owner or operator of the mill must maintain records documenting the source of input parameters including the results of all measurements upon which they are based, the calculations and/or analytical methods used to derive values for input parameters, and the procedure used to determine compliance. In addition, the documentation should be sufficient to allow an independent auditor to verify the accuracy of the determination made concerning the facility's compliance with the standard. These records must be kept at the mill for at least five years and upon request be made available for inspection by the Administrator, or his authorized representative.

§ 61.256 Exemption from the reporting and testing requirements of 40 CFR 61.10.

All facilities designated under this subpart are exempt from the reporting requirements of 40 CFR 61.10.

Subpart X—[Reserved]

Subpart Y—National Emission Standard for Benzene Emissions from Benzene Storage Vessels

SOURCE: 54 FR 38077, Sept. 14, 1989, unless otherwise noted.

§ 61.270 Applicability and designation of sources.

(a) The source to which this subpart applies is each storage vessel that is storing benzene having a specific gravity within the range of specific gravities specified in ASTM D 836-84 for Industrial Grade Benzene, ASTM D 835-85 for Refined Benzene-485, ASTM D 2359-85a for Refined Benzene-535, and ASTM D 4734-87 for Refined Benzene-545. These specifica-

tions are incorporated by reference as specified in § 61.18.

(b) Except for paragraph (b) in § 61.276, storage vessels with a design storage capacity less than 38 cubic meters (10,000 gallons) are exempt from the provisions of this subpart.

(c) This subpart does not apply to storage vessels used for storing benzene at coke by-product facilities.

(d) This subpart does not apply to vessels permanently attached to motor vehicles such as trucks, rail cars, barges, or ships.

(e) This subpart does not apply to pressure vessels designed to operate in excess of 204.9 kPa and without emissions to the atmosphere.

(f) A designated source subject to the provisions of this subpart that is also subject to applicable provisions of 40 CFR part 60 subparts K, Ka, and Kb shall be required to comply only with the subpart that contains the most stringent requirements for that source.

§ 61.271 Emission standard.

The owner or operator of each storage vessel with a design storage capacity greater than or equal to 38 cubic meters (10,000 gallons) to which this subpart applies shall comply with the requirements in paragraph (d) of this section and with the requirements either in paragraph (a), (b), or (c) of this section, or equivalent as provided in § 61.273.

(a) The storage vessel shall be equipped with a fixed roof and an internal floating roof.

(1) An internal floating roof means a cover that rests on the liquid surface (but not necessarily in complete contact with it) inside a storage vessel that has a permanently affixed roof. The internal floating roof shall be floating on the liquid surface at all times, except during initial fill and during those intervals when the storage vessel is completely emptied or subsequently emptied and refilled. When the roof is resting on the leg supports, the process of filling, emptying, or refilling shall be continuous and shall be accomplished as rapidly as possible.

(2) Each internal floating roof shall be equipped with one of the closure

devices listed in paragraphs (a)(2) (i), (ii), or (iii) of this section between the wall of the storage vessel and the edge of the internal floating roof. This requirement does not apply to each existing storage vessel for which construction of an internal floating roof equipped with a continuous seal commenced on or before July 28, 1988. A continuous seal means a seal that forms a continuous closure that completely covers the space between the wall of the storage vessel and the edge of the internal floating roof.

(i) A foam- or liquid-filled seal mounted in contact with the liquid (liquid-mounted seal). A liquid-mounted seal means a foam- or liquid-filled seal mounted in contact with the liquid between the wall of the storage vessel and the floating roof continuously around the circumference of the vessel.

(ii) Two seals mounted one above the other so that each forms a continuous closure that completely covers the space between the wall of the storage vessel and the edge of the internal floating roof. The lower seal may be vapor-mounted, but both must be continuous.

(iii) A metallic shoe seal. A metallic shoe seal (also referred to as a mechanical shoe seal) is, but is not limited to, a metal sheet held vertically against the wall of the storage vessel by springs or weighted levers and is connected by braces to the floating roof. A flexible coated fabric (envelope) spans the annular space between the metal sheet and the floating roof.

(3) Automatic bleeder vents are to be closed at all times when the roof is floating, except when the roof is being floated off or is being landed on the roof leg supports.

(4) Each opening in a noncontact internal floating roof except for automatic bleeder vents (vacuum breaker vents) and the rim space vents is to provide a projection below the liquid surface.

(5) Each internal floating roof shall meet the specifications listed below. If an existing storage vessel had an internal floating roof with a continuous seal as of July 28, 1988, the requirements listed below do not have to be met until the first time after Septem-

ber 14, 1989, the vessel is emptied and degassed or September 14, 1999, whichever occurs first.

(i) Each opening in the internal floating roof except for leg sleeves, automatic bleeder vents, rim space vents, column wells, ladder wells, sample wells, and stub drains is to be equipped with a cover or lid. The cover or lid shall be equipped with a gasket. Covers on each access hatch and automatic gauge float well shall be bolted.

(ii) Each penetration of the internal floating roof for the purposes of sampling shall be a sample well. Each sample well shall have a slit fabric cover that covers at least 90 percent of the opening.

(iii) Each automatic bleeder vent shall be gasketed.

(iv) Rim space vents shall be equipped with a gasket.

(v) Each penetration of the internal floating roof that allows for passage of a ladder shall have a gasketed sliding cover.

(vi) Each penetration of the internal floating roof that allows for passage of a column supporting the fixed roof shall have a flexible fabric sleeve seal or a gasketed sliding cover.

(6) Each cover or lid on any opening in the internal floating roof shall be closed (i.e., no visible gaps), except when a device is in actual use. Covers on each access hatch and each automatic gauge float well which are equipped with bolts shall be bolted when they are not in use. Rim space vents are to be set to open only when the internal floating roof is not floating or at the manufacturer's recommended setting.

(b) The storage vessel shall have an external floating roof.

(1) An external floating roof means a pontoon-type or double-deck-type cover that rests on the liquid surface in a vessel with no fixed roof.

(2) Each external floating roof shall be equipped with a closure device between the wall of the storage vessel and the roof edge. Except as provided in paragraph (b)(5) of this section, the closure device is to consist of two seals, one above the other. The lower seal is referred to as the primary seal and the upper seal is referred to as the secondary seal.

(i) The primary seal shall be either a metallic shoe seal or a liquid-mounted seal. A liquid-mounted seal means a foam- or liquid-filled seal mounted in contact with the liquid between the wall of the storage vessel and the floating roof continuously around the circumference of the vessel. A metallic shoe seal (which can also be referred to as a mechanical shoe seal) is, but is not limited to, a metal sheet held vertically against the wall of the storage vessel by springs or weighted levers and is connected by braces to the floating roof. A flexible coated fabric (envelope) spans the annular space between the metal sheet and the floating roof. Except as provided in § 61.272(b)(4), the primary seal shall completely cover the annular space between the edge of the floating roof and the vessel wall.

(ii) The secondary seal shall completely cover the annular space between the external floating roof and the wall of the storage vessel in a continuous fashion except as allowed in § 61.272(b)(4).

(3) Except for automatic bleeder vents and rim space vents, each opening in the noncontact external floating roof shall provide a projection below the liquid surface. Except for automatic bleeder vents, rim space vents, roof drains, and leg sleeves, each opening in the roof is to be equipped with a gasketed cover, seal or lid which is to be maintained in a closed position at all times (i.e., no visible gap) except when the device is in actual use. Automatic bleeder vents are to be closed at all times when the roof is floating, except when the roof is being floated off or is being landed on the roof leg supports. Rim vents are to be set to open when the roof is being floated off the roof leg supports or at the manufacturer's recommended setting. Automatic bleeder vents and rim space vents are to be gasketed. Each emergency roof drain is to be provided with a slotted membrane fabric cover that covers at least 90 percent of the area of the opening.

(4) The roof shall be floating on the liquid at all times (i.e., off the roof leg supports) except during initial fill until the roof is lifted off leg supports and when the vessel is completely

emptied and subsequently refilled. The process of emptying and refilling when the roof is resting on the leg supports shall be continuous and shall be accomplished as rapidly as possible.

(5) The requirement for a secondary seal does not apply to each existing storage vessel that was equipped with a liquid-mounted primary seal as of July 28, 1988, until after the first time after September 14, 1989, when the vessel is emptied and degassed or 10 years from September 14, 1989, whichever occurs first.

(c) The storage vessel shall be equipped with a closed vent system and a control device.

(1) The closed vent system shall be designed to collect all benzene vapors and gases discharged from the storage vessel and operated with no detectable emissions, as indicated by an instrument reading of less than 500 ppm above background and visual inspections, as determined in § 61.242-11 (Subpart V).

(2) The control device shall be designed and operated to reduce inlet benzene emissions by 95 percent or greater. If a flare is used as the control device, it shall meet the specifications described in the general control device requirements of 40 CFR 60.18.

(3) The specifications and requirements listed in paragraphs (c)(1) and (c)(2) of this section for closed vent systems and control devices do not apply during periods of routine maintenance. During periods of routine maintenance, the benzene level in the storage vessel(s) serviced by the control device subject to the provisions of § 61.271(c) may be lowered but not raised. Periods of routine maintenance shall not exceed 72 hours as outlined in the maintenance plan required by § 61.272(c)(1)(iii).

(4) The specifications and requirements listed in paragraphs (c)(1) and (c)(2) of this section for closed vents and control devices do not apply during a control system malfunction. A control system malfunction means any sudden and unavoidable failure of air pollution control equipment. A failure caused entirely or in part by design deficiencies, poor maintenance, careless operation, or other preventable upset condition or equipment

breakdown is not considered a malfunction.

(d) The owner or operator of each affected storage vessel shall meet the requirements of paragraph (a), (b), or (c) of this section as follows:

(1) The owner or operator of each existing benzene storage vessel shall meet the requirements of paragraph (a), (b), or (c) of this section no later than 90 days after September 14, 1989, with the exceptions noted in paragraphs (a)(5) and (b)(5), unless a waiver of compliance has been approved by the Administrator in accordance with § 61.11.

(2) The owner or operator of each benzene storage vessel upon which construction commenced after September 14, 1989, shall meet the requirements of paragraph (a), (b), or (c) of this section prior to filling (i.e., roof is lifted off leg supports) the storage vessel with benzene.

(3) The owner or operator of each benzene storage vessel upon which construction commenced on or after July 28, 1988, and before September 14, 1989, shall meet the requirements of paragraph (a), (b), or (c) of this section on September 14, 1989.

[54 FR 38077, Sept. 14, 1989; 54 FR 50887, Dec. 11, 1989]

§ 61.272 Compliance provisions.

(a) For each vessel complying with § 61.271(a) (fixed roof and internal floating roof) each owner or operator shall:

(1) After installing the control equipment required to comply with § 61.271(a), visually inspect the internal floating roof, the primary seal, and the secondary seal (if one is in service), prior to filling the storage vessel with benzene. If there are holes, tears or other openings in the primary seal, the secondary seal, or the seal fabric, or defects in the internal floating roof, the owner or operator shall repair the items before filling the storage vessel.

(2) Visually inspect the internal floating roof and the primary seal or the secondary seal (if one is in service) through manholes and roof hatches on the fixed roof at least once every 12 months after initial fill, or at least once every 12 months after September

14, 1989, except as provided in paragraph (a)(4)(i) of this section. If the internal floating roof is not resting on the surface of the benzene liquid inside the storage vessel, or there is liquid on the roof, or the seal is detached, or there are holes or tears in the seal fabric, the owner or operator shall repair the items or empty and remove the storage vessel from service within 45 days. If a failure that is detected during inspections required in this paragraph cannot be repaired within 45 days and if the vessel cannot be emptied within 45 days, an extension of up to 30 additional days may be requested from the Administrator in the inspection report required in § 61.275(a). Such a request for an extension must document that alternate storage capacity is unavailable and specify a schedule of actions the company will take that will ensure that the control equipment will be repaired or the vessel will be emptied as soon as possible.

(3) Visually inspect the internal floating roof, the primary seal, the secondary seal (if one is in service), gaskets, slotted membranes and sleeve seals (if any) each time the storage vessel is emptied and degassed. In no event shall inspections conducted in accordance with this provision occur at intervals greater than 10 years in the case of vessels conducting the annual visual inspections as specified in paragraph (a)(2) of this section and at intervals greater than 5 years in the case of vessels specified in paragraph (a)(4)(i) of this section.

(i) For all the inspections required by paragraphs (a)(1) and (a)(3) of this section, the owner or operator shall notify the Administrator in writing at least 30 days prior to the refilling of each storage vessel to afford the Administrator the opportunity to have an observer present. If the inspection required by paragraph (a)(3) of this section is not planned and the owner or operator could not have known about the inspection 30 days in advance of refilling the vessel, the owner or operator shall notify the Administrator at least 7 days prior to the refilling of the storage vessel. Notification shall be made by telephone immediately followed by written documen-

tation demonstrating why the inspection was unplanned. Alternatively, the notification including the written documentation may be made in writing and sent by express mail so that it is received by the Administrator at least 7 days prior to refilling.

(ii) If the internal floating roof has defects, the primary seal has holes, tears, or other openings in the seal or the seal fabric, or the secondary seal has holes, tears, or other openings in the seal or the seal fabric, or the gaskets no longer close off the liquid surfaces from the atmosphere, or the slotted membrane has more than 10 percent open area, the owner or operator shall repair the items as necessary so that none of the conditions specified in this paragraph exist before refilling the storage vessel with benzene.

(4) For vessels equipped with a double-seal system as specified in § 61.271(a)(2)(ii):

(i) Visually inspect the vessel as specified in paragraph (a)(3) of this section at least every 5 years; or

(ii) Visually inspect the vessel annually as specified in paragraph (a)(2) of this section, and at least every 10 years as specified in paragraph (a)(3) of this section.

(b) For each vessel complying with § 61.271(b) (external floating roof) the owner or operator shall:

(1) Determine the gap areas and maximum gap widths between the primary seal and the wall of the storage vessel, and the secondary seal and the wall of the storage vessel according to the following frequency.

(i) For an external floating roof vessel equipped with primary and secondary seals, measurements of gaps between the vessel wall and the primary seal (seal gaps) shall be performed during the hydrostatic testing of the vessel or within 90 days of the initial fill with benzene or within 90 days of September 14, 1989, whichever occurs last, and at least once every 5 years thereafter, except as provided in paragraph (b)(1)(ii) of this section.

(ii) For an external floating roof vessel equipped with a liquid-mounted primary seal and without a secondary seal as provided for in § 61.271(b)(5), measurement of gaps between the vessel wall and the primary seal (seal

gaps) shall be performed within 90 days of September 14, 1989, and at least once per year thereafter. When a secondary seal is installed over the primary seal, measurement of primary seal gaps shall be performed within 90 days of installation and at least once every 5 years thereafter.

(iii) For an external floating roof vessel equipped with primary and secondary seals, measurements of gaps between the vessel wall and the secondary seal shall be performed within 90 days of the initial fill with benzene, within 90 days of installation of the secondary seal, or within 90 days after September 14, 1989, whichever occurs last, and at least once per year thereafter.

(iv) If any source ceases to store benzene for a period of 1 year or more, subsequent introduction of benzene into the vessel shall be considered an initial fill for the purposes of paragraphs (b)(1)(i), (b)(1)(ii), and (b)(1)(iii) of this section.

(2) Determine gap widths and areas in the primary and secondary seals individually by the following procedures:

(i) Measure seal gaps, if any, at one or more floating roof levels when the roof is floating off the roof leg supports.

(ii) Measure seal gaps around the entire circumference of the vessel in each place where a 0.32 centimeter (cm) (1/8 in) diameter uniform probe passes freely (without forcing or binding against the seal) between the seal and the wall of the storage vessel and measure the circumferential distance of each such location.

(iii) The total surface area of each gap described in paragraph (b)(2)(ii) of this section shall be determined by using probes of various widths to measure accurately the actual distance from the vessel wall to the seal and multiplying each such width by its respective circumferential distance.

(3) Add the gap surface area of each gap location for the primary seal and the secondary seal individually. Divide the sum for each seal by the nominal diameter of the vessel and compare each ratio to the respective standards in § 61.272(b)(4) and § 61.272(b)(5).

(4) Repair conditions that do not meet requirements listed in paragraph

(b)(4) (i) and (ii) within 45 days of identification in any inspection or empty and remove the storage vessel from service within 45 days.

(i) The accumulated area of gaps between the vessel wall and the metallic shoe seal or the liquid-mounted primary seal shall not exceed 212 cm² per meter of vessel diameter (10.0 in² per foot of vessel diameter) and the width of any portion of any gap shall not exceed 3.81 cm (1 1/2 in).

(A) One end of the metallic shoe is to extend into the stored liquid and the other end is to extend a minimum vertical distance of 61 cm (24 in) above the stored liquid surface.

(B) There are to be no holes, tears, or other openings in the shoe, seal fabric, or seal envelope.

(ii) The secondary seal is to meet the following requirements:

(A) The secondary seal is to be installed above the primary seal so that it completely covers the space between the roof edge and the vessel wall except as provided in paragraph (b)(4)(ii)(B) of this section.

(B) The accumulated area of gaps between the vessel wall and the secondary seal shall not exceed 21.2 cm² per meter of vessel diameter (1.0 in² per foot of vessel diameter) or the width of any portion of any gap shall not exceed 1.27 cm (1/2 in). These seal gap requirements may be exceeded during the measurement of primary seal gaps as required by paragraph (b)(1)(i) or (b)(1)(ii) of this section.

(C) There are to be no holes, tears, or other openings in the seal or seal fabric.

(iii) If a failure that is detected during inspections required in this paragraph cannot be repaired within 45 days and if the vessel cannot be emptied within 45 days, an extension of up to 30 additional days may be requested from the Administrator in the inspection report required in § 61.275(d). Such extension request must include a demonstration of unavailability of alternate storage capacity and a specification of a schedule that will assure that the control equipment will be repaired or the vessel will be emptied as soon as possible.

(5) The owner or operator shall notify the Administrator 30 days in

advance of any gap measurements required by paragraph (b)(1) of this section to afford the Administrator the opportunity to have an observer present.

(6) Visually inspect the external floating roof, the primary seal, secondary seal, and fittings each time the vessel is emptied and degassed.

(i) If the external floating roof has defects, the primary seal has holes, tears, or other openings in the seal or the seal fabric, or the secondary seal has holes, tears, or other openings in the seal or the seal fabric, the owner or operator shall repair the items as necessary so that none of the conditions specified in this paragraph exist before filling or refilling the storage vessel with benzene.

(ii) For all the inspections required by paragraph (b)(6) of this section, the owner or operator shall notify the Administrator in writing at least 30 days prior to filling or refilling of each storage vessel to afford the Administrator the opportunity to inspect the storage vessel prior to refilling. If the inspection required by paragraph (b)(6) of this section is not planned and the owner or operator could not have known about the inspection 30 days in advance of refilling the vessel, the owner or operator shall notify the Administrator at least 7 days prior to refilling of the storage vessel. Notification shall be made by telephone immediately followed by written documentation demonstrating why the inspection was unplanned. Alternatively, this notification including the written documentation may be made in writing and sent by express mail so that it is received by the Administrator at least 7 days prior to the refilling.

(c) The owner or operator of each source that is equipped with a closed vent system and control device as required in § 60.271(c), other than a flare, shall meet the following requirements.

(1) Within 90 days after initial fill or after September 14, 1989, whichever comes last, submit for approval by the Administrator, an operating plan containing the information listed below.

(i) Documentation demonstrating that the control device being used achieves the required control efficien-

cy during reasonably expected maximum loading conditions. This documentation is to include a description of the gas stream which enters the control device, including flow and benzene content under varying liquid level conditions (dynamic and static) and manufacturer's design specifications for the control device. If the control device or the closed vent capture system receives vapors, gases or liquids, other than fuels, from sources that are not designated sources under this subpart, the efficiency demonstration is to include consideration of all vapors, gases and liquids received by the closed vent capture system and control device. If an enclosed combustion device with a minimum residence time of 0.75 seconds and a minimum temperature of 816 °C is used to meet the 95 percent requirement, documentation that those conditions exist is sufficient to meet the requirements of this paragraph.

(ii) A description of the parameter or parameters to be monitored to ensure that the control device is operated and maintained in conformance with its design and an explanation of the criteria used for selection of that parameter (or parameters).

(iii) A maintenance plan for the system including the type of maintenance necessary, planned frequency of maintenance, and lengths of maintenance periods for those operations that would require the closed vent system or the control device to be out of compliance with § 61.271(c). The maintenance plan shall require that the system be out of compliance with § 61.271(c) for no more than 72 hours per year.

(2) Operate, monitor the parameters, and maintain the closed vent system and control device in accordance with the operating plan submitted to the Administrator in accordance with paragraph (c)(1) of this section, unless the plan was modified by the Administrator during the approval process. In this case, the modified plan applies.

(d) The owner or operator of each source that is equipped with a closed vent system and a flare to meet the requirements in § 61.271(c) shall meet the requirements as specified in the

general control device requirements in 40 CFR 60.18 (e) and (f).

§ 61.273 Alternative means of emission limitation.

(a) Upon written application from any person, the Administrator may approve the use of alternative means of emission limitation which have been demonstrated to his satisfaction to achieve a reduction in benzene emissions at least equivalent to the reduction in emissions achieved by any requirement in § 61.271 (a), (b), or (c) of this subpart.

(b) Determination of equivalence to the reduction in emissions achieved by the requirements of § 61.271 (a), (b), or (c) will be evaluated using the following information to be included in the written application to the Administrator:

(1) Actual emissions tests that use full-size or scale-model storage vessels that accurately collect and measure all benzene emissions from a given control device, and that accurately simulate wind and account for other emission variables such as temperature and barometric pressure.

(2) An engineering evaluation that the Administrator determines is an accurate method of determining equivalence.

(c) The Administrator may condition approval of equivalency on requirements that may be necessary to ensure operation and maintenance to achieve the same emission reduction as the requirements of § 61.271 (a), (b), or (c).

(d) If, in the Administrator's judgment, an application for equivalence may be approvable, the Administrator will publish a notice of preliminary determination in the *FEDERAL REGISTER* and provide the opportunity for public hearing. After notice and opportunity for public hearing, the Administrator will determine the equivalence of the alternative means of emission limitation and will publish the final determination in the *FEDERAL REGISTER*.

§ 61.274 Initial report.

(a) The owner or operator of each storage vessel to which this subpart applies and which has a design capacity greater than or equal to 38 cubic meters (10,000 gallons) shall submit an

initial report describing the controls which will be applied to meet the equipment requirements in § 61.271. For an existing storage vessel or a new storage vessel for which construction and operation commenced prior to September 14, 1989, this report shall be submitted within 90 days of September 14, 1989, and can be combined with the report required by § 61.10. For a new storage vessel for which construction or operation commenced on or after September 14, 1989, the report shall be combined with the report required by § 61.07. In the case where the owner or operator seeks to comply with § 61.271(c) with a control device other than a flare, this information may consist of the information required by 61.272(c)(1).

(b) The owner or operator of each storage vessel seeking to comply with § 61.271(c) with a flare, shall submit a report containing the measurements required by 40 CFR 60.18(f) (1), (2), (3), (4), (5), and (6). For the owner or operator of an existing storage vessel not seeking to obtain a waiver or a new storage vessel for which construction and operation commenced prior to September 14, 1989, this report shall be combined with the report required by paragraph (a) of this section. For the owner or operator of an existing storage vessel seeking to obtain a waiver, the reporting date will be established in the response to the waiver request. For the owner or operator of a new storage vessel for which construction or operation commenced after September 14, 1989, the report shall be submitted within 90 days of the date the vessel is initially filled (or partially filled) with benzene.

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§ 61.275 Periodic report.

(a) The owner or operator of each storage vessel to which this subpart applies after installing control equipment in accordance with § 61.271(a) (fixed roof and internal floating roof) shall submit a report describing the results of each inspection conducted in accordance with § 61.272(a). For vessels for which annual inspections are

required under § 61.272(a)(2), the first report is to be submitted no more than 12 months after the initial report submitted in accordance with § 61.274, and each report is to be submitted within 60 days of each annual inspection.

(1) Each report shall include the date of the inspection of each storage vessel and identify each storage vessel in which:

(i) The internal floating roof is not resting on the surface of the benzene liquid inside the storage vessel, or there is liquid on the roof, or the seal is detached from the internal floating roof, or there are holes, tears or other openings in the seal or seal fabric; or

(ii) There are visible gaps between the seal and the wall of the storage vessel.

(2) Where an annual report identifies any condition in paragraph (a)(1) of this section the annual report shall describe the nature of the defect, the date the storage vessel was emptied, and the nature of and date the repair was made, except as provided in paragraph (a)(3) of this section.

(3) If an extension is requested in an annual periodic report in accordance with § 61.272(a)(2), a supplemental periodic report shall be submitted within 15 days of repair. The supplemental periodic report shall identify the vessel and describe the date the storage vessel was emptied and the nature of and date the repair was made.

(b) The owner or operator of each storage vessel to which this subpart applies after installing control equipment in accordance with § 61.271(a) (fixed roof and internal floating roof) shall submit a report describing the results of each inspection conducted in accordance with § 61.272(a) (3) or (4).

(1) The report is to be submitted within 60 days of conducting each inspection required by § 61.272(a) (3) or (4).

(2) Each report shall identify each storage vessel in which the owner or operator finds that the internal floating roof has defects, the primary seal has holes, tears, or other openings in the seal or the seal fabric, or the secondary seal (if one has been installed) has holes, tears, or other openings in

the seal or the seal fabric, or the gaskets no longer close off the liquid surfaces from the atmosphere, or the slotted membrane has more than 10 percent open area. The report shall also describe the nature of the defect, the date the storage vessel was emptied, and the nature of and date the repair was made.

(c) Any owner or operator of an existing storage vessel which had an internal floating roof with a continuous seal as of July 28, 1988, and which seeks to comply with the requirements of § 61.271(a)(5) during the first time after September 14, 1989, when the vessel is emptied and degassed but no later than 10 years from September 14, 1989, shall notify the Administrator 30 days prior to the completion of the installation of such controls and the date of refilling of the vessel so the Administrator has an opportunity to have an observer present to inspect the storage vessel before it is refilled. This report can be combined with the one required by § 61.275(b).

(d) The owner or operator of each storage vessel to which this subpart applies after installing control equipment in accordance with § 61.271(b) (external floating roof) shall submit a report describing the results of each seal gap measurement made in accordance with § 61.272(b). The first report is to be submitted no more than 12 months after the initial report submitted in accordance with § 61.274(a), and each annual periodic report is to be submitted within 60 days of each annual inspection.

(1) Each report shall include the date of the measurement, the raw data obtained in the measurement, and the calculations described in § 61.272(b) (2) and (3), and shall identify each storage vessel which does not meet the gap specifications of § 61.272(b). Where an annual report identifies any vessel not meeting the seal gap specifications of § 61.272(b) the report shall describe the date the storage vessel was emptied, the measures used to correct the condition and the date the storage vessel was brought into compliance.

(2) If an extension is requested in an annual periodic report in accordance with § 61.272(b)(4)(iii), a supplemental

periodic report shall be submitted within 15 days of repair. The supplemental periodic report shall identify the vessel and describe the date the vessel was emptied and the nature of and date the repair was made.

(e) Excess emission report.

(1) The owner or operator of each source seeking to comply with § 61.271(c) (vessels equipped with closed vent systems with control devices) shall submit a quarterly report informing the Administrator of each occurrence that results in excess emissions. Excess emissions are emissions that occur at any time when compliance with the specifications and requirements of § 61.271(c) are not achieved, as evidenced by the parameters being measured in accordance with § 61.272(c)(1)(ii) if a control device other than a flare is used, or by the measurements required in § 61.272(d) and the general control device requirements in 40 CFR 60.18(f) (1) and (2) if a flare is used.

(2) The owner or operator shall submit the following information as a minimum in the report required by (e)(1) of this section:

(i) Identify the stack and other emission points where the excess emissions occurred;

(ii) A statement of whether or not the owner or operator believes a control system malfunction has occurred.

(3) If the owner or operator states that a control system malfunction has occurred, the following information as a minimum is also to be included in the report required under paragraph (e)(1) of this section:

(i) Time and duration of the control system malfunction as determined by continuous monitoring data (if any), or the inspections or monitoring done in accordance with the operating plan required by § 61.272(c).

(ii) Cause of excess emissions.

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§ 61.276 Recordkeeping.

(a) Each owner or operator with a storage vessel subject to this subpart shall keep copies of all the reports and records required by this subpart for at least 2 years, except as specified in

paragraphs (b) and (c)(1) of this section.

(b) Each owner or operator with a storage vessel, including any vessel which has a design storage capacity less than 38 cubic meters (10,000 gallons), shall keep readily accessible records showing the dimensions of the storage vessel and an analysis showing the capacity of the storage vessel. This record shall be kept as long as the storage vessel is in operation. Each storage vessel with a design capacity of less than 38 cubic meters (10,000 gallons) is subject to no provisions of this subpart other than those required by this paragraph.

(c) The following information pertaining to closed vent system and control devices shall be kept in a readily accessible location.

(1) A copy of the operating plan. This record shall be kept as long as the closed vent system and control device is in use.

(2) A record of the measured values of the parameters monitored in accordance with § 61.272(c)(1)(ii) and § 61.272(c)(2).

(3) A record of the maintenance performed in accordance with § 61.272(c)(1)(iii) of the operating plan, including the following:

(i) The duration of each time the closed vent system and control device does not meet the specifications of § 61.271(c) due to maintenance, including the following:

(A) The first time of day and date the requirements of 61.271(c) were not met at the beginning of maintenance.

(B) The first time of day and date the requirements of § 61.271(c) were met at the conclusion of maintenance.

(C) A continuous record of the liquid level in each storage vessel that the closed vent system and control device receive vapors from during the interval between the times specified by (c)(3)(1)(A) and (c)(3)(1)(B). Pumping records (simultaneous input and output) may be substituted for records of the liquid level.

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§ 61.277 Delegation of authority.

(a) In delegating implementation and enforcement authority to a State under section 112(d) of the Act, the authorities contained in paragraph (b) of this section shall be retained by the Administrator and not transferred to a State.

(b) Authorities which will not be delegated to States: § 61.273.

Subparts Z-AA—[Reserved]

Subpart BB—National Emission Standard for Benzene Emissions from Benzene Transfer Operations

SOURCE: At 55 FR 8341, Mar. 7, 1990, unless otherwise noted.

§ 61.300 Applicability.

(a) The affected facility to which this subpart applies is the total of all loading racks at which benzene is loaded into tank trucks, railcars, or marine vessels at each benzene production facility and each bulk terminal. However, specifically exempted from this regulation are loading racks at which only the following are loaded: benzene-laden waste (covered under subpart FF of this part), gasoline, or benzene-laden liquid from coke by-product recovery plants.

(b) Any affected facility under paragraph (a) of this section which loads only liquid containing less than 70 weight-percent benzene is exempt from the requirements of this subpart, except for the recordkeeping and reporting requirements in § 61.305(i).

(c) Any affected facility under paragraph (a) of this section shall comply with the standards in § 61.302 at each loading rack that is handling a liquid containing 70 weight-percent or more benzene.

(d) Any affected facility under paragraph (a) of this section whose annual benzene loading is less than 1.3 million liters of 70 weight-percent or more benzene is exempt from the requirements of this subpart, except for the recordkeeping and reporting requirements in § 61.305(i).

(e) The owner or operator of an affected facility, as defined in § 61.300(a)

that loads a marine vessel shall be in compliance with the provisions of this subpart on and after February 28, 1991. If an affected facility that loads a marine vessel also loads a tank truck or railcar, the marine vessel loading racks shall be in compliance with the provisions of this subpart on and after February 28, 1991, while the tank truck loading racks and the railcar loading racks shall be in compliance as required by § 61.12.

§ 61.301 Definitions.

As used in this subpart, all terms not defined herein shall have the meaning given them in the Act, or in subpart A or subpart V of part 61.

Bulk terminal means any facility which receives liquid product containing benzene by pipelines, marine vessels, tank trucks, or railcars, and loads the product for further distribution into tank trucks, railcars, or marine vessels.

Car-sealed means having a seal that is placed on the device used to change the position of a valve (e.g., from open to closed) such that the position of the valve cannot be changed without breaking the seal and requiring the replacement of the old seal, once broken, with a new seal.

Control device means all equipment used for recovering or oxidizing benzene vapors displaced from the affected facility.

Incinerator means any enclosed combustion device that is used for destroying organic compounds and that does not extract energy in the form of steam or process heat. These devices do not rely on the heating value of the waste gas to sustain efficient combustion. Auxiliary fuel is burned in the device and the heat from the fuel flame heats the waste gas to combustion temperature. Temperature is controlled by controlling combustion air or fuel.

Leak means any instrument reading of 10,000 ppmv or greater using method 21 of 40 CFR part 60, appendix A.

Loading cycle means the time period from the beginning of filling a tank truck, railcar, or marine vessel until

flow to the control device ceases, as measured by the flow indicator.

Loading rack means the loading arms, pumps, meters, shutoff valves, relief valves, and other piping and valves necessary to fill tank trucks, railcars, or marine vessels.

Marine vessel means any tank ship or tank barge which transports liquid product such as benzene.

Nonvapor tight means any tank truck, railcar, or marine vessel that does not pass the required vapor-tightness test.

Process heater means a device that transfers heat liberated by burning fuel to fluids contained in tubes, except water that is heated to produce steam.

Steam generating unit means any enclosed combustion device that uses fuel energy in the form of steam.

Vapor collection system means any equipment located at the affected facility used for containing benzene vapors displaced during the loading of tank trucks, railcars, or marine vessels. This does not include the vapor collection system that is part of any tank truck, railcar, or marine vessel vapor collection manifold system.

Vapor-tight marine vessel means a marine vessel with a benzene product tank that has been demonstrated within the preceding 12 months to have no leaks. This demonstration shall be made using method 21 of part 60, appendix A, during the last 20 percent of loading and during a period when the vessel is being loaded at its maximum loading rate. A reading of greater than 10,000 ppm as methane shall constitute a leak. As an alternative, a marine vessel owner or operator may use the vapor-tightness test described in § 61.304(f) to demonstrate vapor tightness. A marine vessel operated at negative pressure is assumed to be vapor-tight for the purpose of this standard.

Vapor-tight tank truck or vapor-tight railcar means a tank truck or railcar for which it has been demonstrated within the preceding 12 months that its product tank will sustain a pressure change of not more than 750 pascals within 5 minutes after it is pressurized to a minimum of 4,500 pascals. This capability is to be

demonstrated using the pressure test procedure specified in method 27 of part 60, appendix A, and a pressure measurement device which has a precision of ± 2.5 mm water and which is capable of measuring above the pressure at which the tank truck or railcar is to be tested for vapor tightness.

§ 61.302 Standards.

(a) The owner or operator of an affected facility shall equip each loading rack with a vapor collection system that is:

(1) Designed to collect all benzene vapors displaced from tank trucks, railcars, or marine vessels during loading, and

(2) Designed to prevent any benzene vapors collected at one loading rack from passing through another loading rack to the atmosphere.

(b) The owner or operator of an affected facility shall install a control device and reduce benzene emissions routed to the atmosphere through the control device by 98 weight percent. If a boiler or process heater is used to comply with the percent reduction requirement, then the vent stream shall be introduced into the flame zone of such a device.

(c) The owner or operator of an affected facility shall operate any flare used to comply with paragraph (b) of this section in accordance with the requirements of § 60.18 (b) through (f).

(d) The owner or operator of an affected facility shall limit loading of benzene into vapor-tight tank trucks and vapor-tight railcars using the following procedures:

(1) The owner or operator shall obtain the vapor-tightness documentation described in § 61.305(h) for each tank truck or railcar loaded at the affected facility. The test date in the documentation must be within the preceding 12 months. The vapor-tightness test to be used for tank trucks and railcars is method 27 of part 60, appendix A.

(2) The owner or operator shall cross-check the identification number for each tank truck or railcar to be loaded with the file of vapor-tightness documentation before the corresponding tank truck or railcar is loaded. If

no documentation is on file, the owner or operator shall obtain a copy of the information from the tank truck or railcar operator before the tank truck or railcar is loaded.

(3) Alternate procedures to those described in paragraphs (d)(1) and (d)(2) of this section may be used upon application to, and approval by, the Administrator.

(e) The owner or operator of an affected facility shall limit the loading of marine vessels to those vessels that are vapor tight as determined by either paragraph (e)(1), (e)(2), (e)(3), or (e)(4) of this section.

(1) The owner or operator of an affected facility shall ensure that each marine vessel is loaded with the benzene product tank below atmospheric pressure (i.e., at negative pressure). If the pressure is measured at the interface between the shoreside vapor collection pipe and the marine vessel vapor line, the pressure measured according to the procedures in § 61.303(f) must be below atmospheric pressure.

(2) The owner or operator of an affected facility shall use the following procedure to obtain the vapor-tightness documentation described in § 61.305(h). The vapor-tightness test for marine vessels is method 21 of part 60, appendix A, and shall be applied to any potential sources of vapor leaks. A reading of 10,000 ppmv or greater as methane shall constitute a leak.

(i) The owner or operator of an affected facility shall obtain the leak test documentation described in § 61.305(h) for each marine vessel prior to loading, if available. The date of the test listed in the documentation must be within the 12 preceding months.

(ii) If there is no documentation of a successful leak test conducted on the marine vessel in the preceding 12 months, the owner or operator of an affected facility shall require that a leak test of the marine vessel be conducted during the final 20 percent of loading of the marine vessel or shall not load the vessel. The test shall be conducted when the marine vessel is being loaded at the maximum allowable loading rate.

(A) If no leak is detected, the owner or operator of an affected facility shall require that the documentation described in § 61.305(h) is completed prior to departure of the vessel. The owner or operator of the affected facility shall retain a copy of the vapor-tightness documentation on file.

(B) If any leak is detected, the owner or operator of an affected facility shall require that the vapor-tightness failure be documented for the marine vessel owner or operator prior to departure of the vessel. The owner or operator of the affected facility shall retain a copy of the vapor-tightness documentation on file. Delay of repair of equipment for which leaks have been detected will be allowed if the repair is technically infeasible without dry-docking the vessel. This equipment will be excluded from future method 21 tests until repairs are effected. Repair of this equipment shall occur the next time the vessel is dry-docked.

(iii) If the marine vessel has failed its most recent vapor-tightness test as described in § 61.302(e)(2)(ii), the owner or operator of the affected facility shall require that the owner or operator of the nonvapor-tight marine vessel provide documentation that the leaks detected during the previous vapor-tightness test have been repaired, or proof that repair is technically infeasible without dry-docking the vessel. Once the repair documentation has been provided, the owner or operator may load the marine vessel. The owner or operator shall require that the vapor-tightness test described in § 61.302(e)(2)(ii) be conducted during loading, and shall retain a copy of the vapor-tightness documentation on file.

(3) The owner or operator of an affected facility shall obtain a copy of the marine vessel's vapor-tightness documentation described in § 61.305(h) for a test conducted within the preceding 12 months in accordance with § 61.304(f).

(4) Alternate procedures to those described in paragraphs (e)(1), (e)(2) and (e)(3) of this section may be used upon application to, and approval by, the Administrator.

(f) The owner or operator of an affected facility shall limit loading of benzene to tank trucks, railcars, and marine vessels equipped with vapor collection equipment that is compatible with the affected facility's vapor collection system.

(g) The owner or operator of an affected facility shall limit loading of tank trucks, railcars, and marine vessels to tank trucks, railcars, and marine vessels whose collection systems are connected to the affected facility's vapor collection systems.

(h) The owner or operator of an affected facility shall ensure that the vapor collection and benzene loading equipment of tank trucks and railcars shall be designed and operated to prevent gauge pressure in the tank truck or railcar tank from exceeding, during loading, the initial pressure the tank was pressured up to and shown to be vapor tight at during the most recent vapor-tightness test using method 27 of part 60, appendix A. This vapor-tightness test pressure is not to be exceeded when measured by the procedures specified in § 61.304(c).

(i) The owner or operator of an affected facility shall ensure that no pressure-vacuum vent in the affected facility's vapor collection system for tank trucks and railcars shall begin to open at a system pressure less than the maximum pressure at which the tank truck or railcar is operated.

(j) The owner or operator of an affected facility shall ensure that the maximum normal operating pressure of the marine vessel's vapor collection equipment shall not exceed 0.8 times the relief set pressure of the pressure-vacuum vents. This level is not to be exceeded when measured by the procedures specified in § 61.304(d).

(k) The owner or operator of an affected facility shall inspect the vapor collection system and the control device for detectable emissions, and shall repair any leaks detected, in accordance with § 61.242-11 (e) and (f). This inspection of the vapor collection system and control device shall be done during the loading of tank trucks, railcars, or marine vessels.

(l) Vent systems that contain valves that could divert a vent stream from a control device shall have car-sealed

opened all valves in the vent system from the emission source to the control device, and car-sealed closed all valves in the vent system that would lead the vent stream to the atmosphere, either directly or indirectly, by-passing the control device.

§ 61.363 Monitoring requirements.

(a) Each owner or operator of an affected facility that uses an incinerator to comply with the percent reduction requirement specified under § 61.302(b) shall install, calibrate, maintain, and operate according to manufacturer's specifications a temperature monitoring device equipped with a continuous recorder and having an accuracy of ± 1 percent of the combustion temperature being measured expressed in degrees Celsius or $\pm 0.5^\circ\text{C}$, whichever is greater.

(1) Where an incinerator other than a catalytic incinerator is used, the owner or operator of the affected facility shall install a temperature monitoring device in the firebox.

(2) Where a catalytic incinerator is used, the owner or operator shall install temperature monitoring devices in the gas stream immediately before and after the catalyst bed.

(b) Each owner or operator of an affected facility that uses a flare to comply with § 61.302(b) shall install, calibrate, maintain, and operate according to manufacturer's specifications a heat sensing device, such as an ultraviolet beam sensor or thermocouple, at the pilot light to indicate the presence of a flame during the entire loading cycle.

(c) Each owner or operator of an affected facility that uses a steam generating unit or process heater to comply with § 61.302(b) shall comply with the following requirements. Where a steam generating unit with a design heat input capacity of less than 44 MW is used to comply with § 61.302(b), the owner or operator of an affected facility shall comply with paragraph (c)(1) of this section. Where a steam generating unit or process heater with a design heat input capacity of 44 MW or greater is used to comply with § 61.302(b), the owner or operator of

an affected facility shall comply with paragraph (c)(2) of this section.

(1) Install in the firebox, calibrate, maintain, and operate according to manufacturer's specifications a temperature monitoring device equipped with a continuous recorder and having an accuracy of ± 1 percent of the temperature being measured expressed in degrees Celsius or $\pm 0.5^\circ\text{C}$, whichever is greater, for steam generating units or process heaters of less than 44 MW design heat input capacity.

(2) Monitor and record the periods of operation of the steam generating units or process heater if the design heat input capacity of the steam generating unit or process heater is 44 MW or greater. The records must be readily available for inspection.

(d) Each owner or operator of an affected facility that uses a carbon adsorption system to comply with the percent reduction requirement specified under § 61.302(b) shall install, calibrate, maintain, and operate according to manufacturer's specifications a device that continuously indicates and records the concentration or reading of organic compounds in the outlet gas stream of each carbon adsorber bed.

(e) The owner or operator of an affected facility who wishes to demonstrate compliance with the standards specified under § 61.302(b) using control devices other than an incinerator, steam generating unit, process heater, carbon adsorber, or flare shall provide the Administrator with information describing the operation of the control device and the process parameter(s) that would indicate proper operation and maintenance of the device. The Administrator may request further information and will specify appropriate monitoring procedures or requirements.

(f) Each owner or operator of an affected facility complying with § 61.302(e)(1) shall install, calibrate, maintain, and operate a recording pressure measurement device (magnetic gauge or equivalent device) and an audible and visible alarm system that is activated when the pressure vacuum specified in § 61.302(e)(1) is not attained. The owner or operator shall place the alarm system so that it

can be seen and heard where cargo transfer is controlled and on the open deck.

(g) Owners or operators using a vent system that contains valves that could divert a vent stream from a control device used to comply with the provisions of this subpart shall do one or a combination of the following:

(1) Install a flow indicator immediately downstream of each valve that if opened would allow a vent stream to bypass the control device and be emitted, either directly or indirectly, to the atmosphere. The flow indicator shall be capable of recording flow at least once every 15 minutes.

(2) Monitor the valves once a month, checking the position of the valves and the condition of the car seal, and identify all times when the car seals have been broken and the valve position has been changed (i.e., from opened to closed for valves in the vent piping to the control device and from closed to open for valves that allow the stream to be vented directly or indirectly to the atmosphere).

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§ 61.304 Test methods and procedures.

(a) The procedures for determining compliance with § 61.302(b) for all control devices other than flares is as follows:

(1) All testing equipment shall be prepared and installed as specified in the appropriate test methods.

(2) The time period for a performance test shall be not less than 6 hours, during which at least 300,000 liters of benzene are loaded. If the throughput criterion is not met during the initial 6 hours, the test may be either continued until the throughput criterion is met, or resumed the next day with at least another 6 complete hours of testing.

(3) For intermittent control devices:

(i) The vapor holder level of the intermittent control device shall be recorded at the start of the performance test. The end of the performance test shall coincide with the time when the vapor holder is at its original level.

(ii) At least two startups and shutdowns of the control device shall occur during the performance test. If this does not occur under an automatically controlled operation, the system shall be manually controlled.

(4) An emission testing interval shall consist of each 5-minute period during the performance test. For each interval:

(i) The reading from each measurement instrument shall be recorded.

(ii) Method 1 or 1A of part 60, appendix A, as appropriate, shall be used for selection of the sampling site.

(iii) The volume exhausted shall be determined using method 2, 2A, 2C, or 2D of part 60, appendix A, as appropriate.

(iv) The average benzene concentration upstream and downstream of the control device in the vent shall be determined using method 25A or method 25B of appendix A of this part, using benzene as the calibration gas. The average benzene concentration shall correspond to the volume measurement by taking into account the sampling system response time.

(5) The mass emitted during each testing interval shall be calculated as follows:

$$M_i = FKV_s C$$

where:

M_i = Mass of benzene emitted during testing interval i , kg.

V_s = Volume of air-vapor mixture exhausted, m^3 at standard conditions.

C = Benzene concentration (as measured) at the exhaust vent, ppmv.

K = Density, (kg/ m^3 benzene), standard conditions.

K = 3.25 for benzene.

F = Conversion factor, (m^3 benzene/ m^3 air)(1/ppmv).

F = 10/ ρ , ρ = density of air at standard conditions.

s = Standard conditions, 20 °C and 760 mm Hg.

(6) The benzene mass emission rates before and after the control device shall be calculated as follows:

$$E = \frac{\sum_{i=1}^n M_i}{T}$$

where:

E = Mass flow rate of benzene emitted, kg/hr.

M_i = Mass of benzene emitted during testing interval i , kg.

T = Total time of all testing intervals, hr.

n = Number of testing intervals.

(7) The percent reduction across the control device shall be calculated as follows:

$$R = \frac{E_b - E_a}{E_b} (100)$$

where:

R = Control efficiency of control device, %.

E_b = Mass flow rate of benzene prior to control device, kg/hr.

E_a = Mass flow rate of benzene after control device, kg/hr.

(b) When a flare is used to comply with § 61.302(b), a performance test according to method 22 of appendix A of this part, shall be performed to determine visible emissions. The observation period shall be at least 2 hours and shall be conducted according to method 22. Performance testing shall be conducted during at least three complete loading cycles with a separate test run for each loading cycle. The observation period for detecting visible emissions shall encompass each loading cycle. Integrated sampling to measure process vent stream flow rate shall be performed continuously during each loading cycle.

(c) For the purpose of determining compliance with § 61.302(h), the following procedures shall be used:

(1) Calibrate and install a pressure measurement device (liquid manometer, magnehelic gauge, or equivalent instrument), which has a precision of ± 2.5 mm H₂O in the range that the tank truck or railcar was initially pressured to during the most recent vapor-tightness test.

(2) Connect the pressure measurement device to a pressure tap in the affected facility's vapor collection system, located as close as possible to the connection with the tank truck or railcar.

(3) During the performance test, record the pressure every 5 minutes while a tank truck or railcar is being

loaded, and record the highest instantaneous pressure that occurs during each loading cycle. Every loading rack shall be tested at least once during the performance test.

(4) If more than one loading rack is used simultaneously, then the performance test shall be conducted simultaneously to represent the maximum capacity.

(d) For the purpose of determining compliance with § 61.302(j), the following procedures shall be used:

(1) Calibrate and install a pressure measurement device (liquid manometer, magnehelic gauge, or equivalent instrument), capable of measuring up to the relief set pressure of the pressure-vacuum vents.

(2) Connect the pressure measurement device to a pressure tap in the affected facility's vapor collection system, located as close as possible to the connection with the marine vessel.

(3) During the performance test, record the pressure every 5 minutes while a marine vessel is being loaded, and record the highest instantaneous pressure that occurs during each loading cycle.

(e) Immediately prior to a performance test required for determination of compliance with § 61.302(b), all potential sources of vapor leakage in the affected facility's vapor collection system equipment shall be inspected for detectable emissions as required in § 61.302(k). The monitoring shall be conducted only while a vapor-tight tank truck, railcar, or marine vessel is being loaded. All identified leaks in the terminal's vapor collection system shall be repaired prior to conducting the performance test.

(f) The following test method shall be used to comply with the marine vessel vapor-tightness requirements of § 61.302(e)(3):

(1) Each benzene product tank shall be pressurized with dry air or inert gas to not less than 1.0 psig and not more than the pressure of the lowest relief valve setting.

(2) Once the pressure is obtained, the dry air or inert gas source shall be shut off.

(3) At the end of one-half hour, the pressure in the benzene product tank and piping shall be measured. The

change in pressure shall be calculated using the following formula:

$$\Delta P = P_1 - P_2$$

where:

ΔP = Change in pressure, inches of water.

P_1 = Pressure in tank when air/gas source is shut off, inches of water.

P_2 = Pressure in tank at the end of one-half hour after air/gas source is shut off, inches of water.

(4) The change in pressure, ΔP , shall be compared to the pressure drop calculated using the following formula:

$$\Delta P_M = 0.861 P_a L/V$$

where:

ΔP_M = Maximum allowable pressure change, inches of water.

P_a = Pressure in tank when air/gas source is shut off, pounds per square inch, absolute (psia).

L = Maximum permitted loading rate of vessel, barrels per hour.

V = Total volume of product tank, barrels.

(5) If $\Delta P < \Delta P_M$, the vessel is vapor tight.

(6) If $\Delta P > \Delta P_M$, the vessel is not vapor tight and the source of the leak must be identified and repaired prior to retesting.

[55 FR 8341, Mar. 7, 1990; 55 FR 12444, Apr. 3, 1990]

§ 61.305 Reporting and recordkeeping.

(a) Each owner or operator of an affected facility subject to the provisions of this subpart shall keep an up-to-date, readily accessible record of the following data measured during each performance test, and also include the following data in the report of the initial performance test required under § 61.13. Where a steam generating unit or process heater with a design heat input capacity of 44 MW or greater is used to comply with § 61.302(b), a report containing performance test data need not be submitted, but a report containing the information in § 61.305(a)(3)(i) is required.

(1) Where an owner or operator subject to the provisions of this subpart is complying with § 61.302(b) through use of an incinerator:

(i) The average firebox temperature of the incinerator (or the average temperature upstream and downstream of the catalyst bed), measured at least every 2 minutes during a loading cycle if the total time period of the loading

cycle is less than 3 hours and every 15 minutes if the total time period of the loading cycle is equal to or greater than 3 hours. The measured temperature shall be averaged over the loading cycle.

(ii) The percent reduction of benzene determined as specified in § 61.304(a) achieved by the incinerator.

(iii) The duration of the loading cycle.

(2) Where an owner or operator subject to the provisions of this subpart is complying with § 61.302 (b) and (c) through use of a smokeless flare or other flare design (i.e., steam-assisted, air-assisted or nonassisted), all visible emission readings, heat content determination, flow rate measurements, maximum permitted velocity calculations, and exit velocity determinations made during the performance test, continuous records of the flare pilot flame monitoring measured continuously during the loading cycle, duration of all loading cycles and records of all loading cycles during which the pilot flame is absent for each vent stream.

(3) Where an owner or operator subject to the provisions of this subpart is complying with § 61.302(b) through the use of a steam generating unit or process heater:

(i) A description of the location at which the vent stream is introduced into the steam generating unit or process heater.

(ii) The average combustion temperature of the steam generating unit or process heater with a design heat input capacity of less than 44 MW measured at least every 2 minutes during a loading cycle if the total time period of the loading cycle is less than 3 hours and every 15 minutes if the total time period of the loading cycle is equal to or greater than 3 hours. The measured temperature shall be averaged over the loading cycle.

(iii) The duration of the loading cycle.

(4) Where an owner or operator subject to the provisions of this subpart is complying with § 61.302(b) through the use of a carbon adsorption system, the control efficiency, R , of the carbon adsorption system, and all sup-

porting performance test data and calculations used to determine that value.

(5) Each owner or operator subject to the provisions of this subpart shall submit with the initial performance test an engineering report describing in detail the vent system used to vent each affected vent stream to a control device. This report shall include all valves and vent pipes that could vent the stream to the atmosphere, thereby bypassing the control device, and identify which valves are car-sealed opened and which valves are car-sealed closed.

(b) Each owner or operator subject to the provisions of this subpart shall keep up-to-date, readily accessible continuous records of the equipment operating parameters specified to be monitored under § 61.303 (a), (c), and (d) as well as up-to-date, readily accessible records of periods of operation during which the parameter boundaries established during the most recent performance test are exceeded. The Administrator may at any time require a report of these data. Periods of operation during which the parameter boundaries established during the most recent performance tests are exceeded are defined as follows:

(1) For thermal incinerators, all loading cycles during which the average combustion temperature was more than 28°C below the average loading cycle combustion temperature during the most recent performance test at which compliance with § 61.302(b) was determined.

(2) For catalytic incinerators, all loading cycles during which the average temperature of the vent stream immediately before the catalyst bed is more than 28°C below the average temperature of the process vent stream during loading cycles during the most recent performance test at which compliance with § 61.302(b) was determined.

(3) All loading cycles during which the average combustion temperature was more than 28°C below the average combustion temperature during the most recent performance test at which compliance with § 61.302(b) was determined for steam generating units or process heaters with a design heat input capacity of less than 44 MW.

(4) For steam generating units or process heaters, whenever there is a change in the location at which the vent stream is introduced into the flame zone as required under § 61.302(b).

(5) For carbon adsorbers, all 3-hour periods of operation during which the average VOC concentration or reading of organics in the exhaust gases is more than 20 percent greater than the average exhaust gas concentration or reading measured by the organics monitoring device during the most recent determination of the recovery efficiency of the carbon adsorber that demonstrated that the facility was in compliance.

(c) If a vent system containing valves that could divert the emission stream away from the control device is used, each owner or operator subject to the provisions of this subpart shall keep for at least 2 years up-to-date, readily accessible continuous records of:

(1) All periods when flow is indicated if flow indicators are installed under § 61.303(g)(1).

(2) All times when maintenance is performed on car-sealed valves, when the car seal is broken, and when the valve position is changed (i.e., from open to closed for valves in the vent piping to the control device and from closed to open for valves that vent the stream directly or indirectly to the atmosphere bypassing the control device) if valves are monitored under § 60.303(g)(2).

(d) Each owner or operator of an affected facility subject to the provisions of this subpart who uses a steam generating unit or process heater with a design heat input capacity of 44 MW or greater to comply with § 61.302(b) shall keep an up-to-date, readily accessible record of all periods of operation of the steam generating unit or process heater. Examples of such records could include records of steam use, fuel use, or monitoring data collected pursuant to other State or Federal regulatory requirements.

(e) Each owner or operator of an affected facility subject to the provisions of this subpart shall keep up-to-date, readily accessible records of the flare pilot flame monitoring specified under § 61.303(b), as well as up-to-date, read-

ily accessible records of any absence of the pilot flame during a loading cycle.

(f) Each owner or operator of an affected facility subject to the requirements of § 61.302 shall submit to the Administrator quarterly reports of the following information. The owner or operator shall submit the initial report within 90 days after the effective date of this subpart or 90 days after startup for a source that has an initial startup date after the effective date.

(1) Periods of operation where there were exceedances of monitored parameters recorded under § 61.305(b).

(2) All periods recorded under § 61.305(c)(1) when the vent stream is diverted from the control device.

(3) All periods recorded under § 61.305(d) when the steam generating unit or process heater was not operating.

(4) All periods recorded under § 61.305(e) in which the pilot flame of the flare was absent.

(5) All times recorded under § 61.305(c)(2) when maintenance is performed on car-sealed valves, when the car seal is broken, and when the valve position is changed.

(g) The owner or operator of an affected facility shall keep the vapor-tightness documentation required under § 61.302 (d) and (e) on file at the affected facility in a permanent form available for inspection.

(h) The owner or operator of an affected facility shall update the documentation file required under § 61.302 (d) and (e) for each tank truck, railcar, or marine vessel at least once per year to reflect current test results as determined by the appropriate method. The owner or operator shall include, as a minimum, the following information in this documentation:

(1) Test title;

(2) Tank truck, railcar, or marine vessel owner and address;

(3) Tank truck, railcar, or marine vessel identification number;

(4) Testing location;

(5) Date of test;

(6) Tester name and signature;

(7) Witnessing inspector: name, signature, and affiliation; and

(8) Test results, including, for railcars and tank trucks, the initial pres-

sure up to which the tank was pressured at the start of the test.

(i) Each owner or operator of an affected facility complying with § 61.300(b) or § 61.300(d) shall record the following information. The first year after promulgation the owner or operator shall submit a report containing the requested information to the Director of the Emission Standards Division, (MD-13), U.S. Environmental Protection Agency, Research Triangle Park, North Carolina 27711. After the first year, the owner or operator shall continue to record; however, no reporting is required. The information shall be made available if requested. The information shall include, as a minimum:

- (1) The affected facility's name and address;
- (2) The weight percent of the benzene loaded;
- (3) The type of vessel loaded (i.e., tank truck, railcar, or marine vessel); and
- (4) The annual amount of benzene loaded into each type of vessel.

(Approved by the Office of Management Budget under control number 2060-0182)

§ 61.306 Delegation of authority.

(a) In delegating implementation and enforcement authority to a State under section 112(d) of the Act, the authorities contained in paragraph (b) of this section shall be retained by the Administrator and not transferred to a State.

(b) Authorities which will not be delegated to States: No restrictions.

Subparts CC-EE—[Reserved]

Subpart FF—National Emission Standard for Benzene Waste Operations

SOURCE: 55 FR 8346, Mar. 7, 1990, unless otherwise noted.

§ 61.340 Applicability.

(a) The provisions of this subpart apply to owners and operators of chemical manufacturing plants, coke by-product recovery plants, and petroleum refineries.

(b) The provisions of this subpart apply to owners and operators of facilities at which waste management units are used to treat, store, or dispose of waste generated by any facility listed in paragraph (a) of this section.

(c) At each facility identified in paragraph (a) or (b) of this section, the following waste is exempt from the requirements of this subpart:

- (1) Waste in the form of gases or vapors that is emitted from process fluids;
- (2) Waste that is contained in a segregated stormwater sewer system; and
- (3) Waste that is not discharged from the process unit which generates the waste stream and, instead, is returned directly to the process. Examples of such waste are intermediate and product distillation reflux streams.

§ 61.341 Definitions.

Benzene concentration means the fraction by weight of benzene in a waste as determined in accordance with the procedures specified in § 61.355 of this subpart.

Chemical manufacturing plant means any facility engaged in the production of chemicals by chemical, thermal, physical, or biological processes for use as a product, co-product, by-product, or intermediate including but not limited to industrial organic chemicals, organic pesticide products, pharmaceutical preparations, paint and allied products, fertilizers, and agricultural chemicals. Examples of chemical manufacturing plants include facilities at which process units are operated to produce one or more of the following chemicals: benzene-sulfonic acid, benzene, chlorobenzene, cumene, cyclohexane, ethylene, ethylbenzene, hydroquinone, linear alkylbenzene, nitrobenzene, resorcinol, sulfonolane, or styrene.

Closed-vent system means a system that is not open to the atmosphere and is composed of piping, ductwork, connections, and, if necessary, flow inducing devices that transport gas or vapor from an emission source to a control device.

Coke by-product recovery plant means any facility designed and oper-

ated for the separation and recovery of coal tar derivatives (by-products) evolved from coal during the coking process of a coke oven battery.

Container means any portable waste management unit in which a material is stored, transported, treated, or otherwise handled. Examples of containers are drums, barrels, tank trucks, barges, dumpsters, tank cars, dump trucks, and ships.

Control device means an enclosed combustion device, vapor recovery system, or flare.

Cover means a device or system which is placed on or over a waste placed in a waste management unit so that the entire waste surface area is enclosed and sealed to minimize air emissions. A cover may have openings necessary for operation, inspection, and maintenance of the waste management unit such as access hatches, sampling ports, and gauge wells provided that each opening is closed and sealed when not in use. Example of covers include a fixed roof installed on a tank, a lid installed on a container, and an air-supported enclosure installed over a waste management unit.

External floating roof means a pontoon-type or double-deck type cover with certain rim sealing mechanisms that rests on the liquid surface in a waste management unit with no fixed roof.

Facility means all process units and product tanks that generate waste within a stationary source, and all waste management units that are used for waste treatment, storage, or disposal within a stationary source.

Fixed roof means a cover that is mounted on a waste management unit in a stationary manner and that does not move with fluctuations in liquid level.

Floating roof means a cover with certain rim sealing mechanisms consisting of a double deck, pontoon single deck, internal floating cover or covered floating roof, which rests upon and is supported by the liquid being contained, and is equipped with a closure seal or seals to close the space between the roof edge and unit wall.

Individual drain system means the system used to convey waste from a

process unit, product storage tank, or waste management unit to a waste management unit. The term includes all process drains and common junction boxes, together with their associated sewer lines and other junction boxes, down to the receiving waste management unit.

Internal floating roof means a cover that rests or floats on the liquid surface inside a waste management unit that has a fixed roof.

Liquid-mounted seal means a foam or liquid-filled primary seal mounted in contact with the liquid between the waste management unit wall and the floating roof continuously around the circumference.

Loading means the introduction of waste into a waste management unit but not necessarily to complete capacity (also referred to as filling).

No detectable emissions means less than 500 parts per million by volume (ppmv) above background levels, as measured by a detection instrument reading in accordance with the procedures specified in § 61.355(h) of this subpart.

Oil-water separator means a waste management unit, generally a tank or surface impoundment, used to separate oil from water. An oil-water separator consists of not only the separation unit but also the forebay and other separator basins, skimmers, weirs, grit chambers, sludge hoppers, and bar screens that are located directly after the individual drain system and prior to additional treatment units such as an air flotation unit, clarifier, or biological treatment unit. Examples of an oil-water separator include an API separator, parallel-plate interceptor, and corrugated-plate interceptor with the associated ancillary equipment.

Petroleum refinery means any facility engaged in producing gasoline, kerosene, distillate fuel oils, residual fuel oils, lubricants, or other products through the distillation of petroleum, or through the redistillation, cracking, or reforming of unfinished petroleum derivatives.

Petroleum means the crude oil removed from the earth and the oils derived from tar sands, shale, and coal.

Point of waste generation means the location where samples of a waste stream are collected for the purpose of determining the waste flow rate, water content, or benzene concentration in accordance with procedures specified in § 61.355 of this subpart. For a chemical manufacturing plant or petroleum refinery, the point of waste generation is a location after the waste stream exits the process unit component, product tank, or waste management unit generating the waste, and before the waste is exposed to the atmosphere or mixed with other wastes. For a coke-by-product recovery plant subject to and complying with the control requirements of §§ 61.132, 61.133, or 61.134 of this part, the point of waste generation is a location after the waste stream exits the process unit component or waste management unit controlled by that subpart, and before the waste is exposed to the atmosphere. For other facilities subject to this subpart, the point of waste generation is a location after the waste enters the facility, and before the waste is exposed to the atmosphere or placed in a facility waste management unit.

Process unit means equipment assembled and connected by pipes or ducts to produce intermediate or final products. A process unit can be operated independently if supplied with sufficient fuel or raw materials and sufficient product storage facilities.

Process wastewater means water which comes in contact with benzene during manufacturing or processing operations conducted within a process unit. Process wastewater is not organic wastes, process fluids, product tank drawdown, cooling tower blowdown, steam trap condensate, or landfill leachate.

Process wastewater stream means a waste stream that contains only process wastewater.

Product tank means a stationary unit that is designed to contain an accumulation of materials that are fed to or produced by a process unit, and is constructed primarily of non-earth materials (e.g., wood, concrete, steel, plastic) which provide structural support.

Product tank drawdown means any material or mixture of materials discharged from a product tank for the purpose of removing water or other contaminants from the product tank.

Segregated stormwater sewer system means a drain and collection system designed and operated for the sole purpose of collecting rainfall runoff at a facility, and which is segregated from all other individual drain systems.

Sewer line means a lateral, trunk line, branch line, or other enclosed conduit used to convey waste to a downstream waste management unit.

Slop oil means the floating oil and solids that accumulate on the surface of an oil-water separator.

Surface impoundment means a waste management unit which is a natural topographic depression, man-made excavation, or diked area formed primarily of earthen materials (although it may be lined with man-made materials), which is designed to hold an accumulation of liquid wastes or waste containing free liquids, and which is not an injection well. Examples of surface impoundments are holding, storage, settling, and aeration pits, ponds, and lagoons.

Tank means a stationary waste management unit that is designed to contain an accumulation of waste and is constructed primarily of non-earth materials (e.g., wood, concrete, steel, plastic) which provide structural support.

Treatment process means a stream stripping unit, thin-film evaporation unit, waste incinerator, or any other process used to comply with § 61.348 of this subpart.

Vapor-mounted seal means a foam-filled primary seal mounted continuously around the perimeter of a waste management unit so there is an annular vapor space underneath the seal. The annular vapor space is bounded by the bottom of the primary seal, the unit wall, the liquid surface, and the floating roof.

Waste means any material resulting from industrial, commercial, mining or agricultural operations, or from community activities that is discarded or is being accumulated, stored, or physically, chemically, thermally, or biologically.

cally treated prior to being discarded, recycled, or discharged.

Waste management unit means a piece of equipment, structure, or transport mechanism used in handling, storage, treatment, or disposal of waste. Examples of a waste management unit include a tank, surface impoundment, container, oil-water separator, individual drain system, steam stripping unit, thin-film evaporation unit, waste incinerator, and landfill.

Waste stream means the waste generated by a particular process unit, product tank, or waste management unit. The characteristics of the waste stream (e.g., flow rate, benzene concentration, water content) are determined at the point of waste generation. Examples of a waste stream include process wastewater, product tank drawdown, sludge and slop oil removed from waste management units, and landfill leachate.

Wastewater treatment system means any component, piece of equipment, or installation that receives, manages, or treats process wastewater, product tank drawdown, or landfill leachate prior to direct or indirect discharge in accordance with the National Pollutant Discharge Elimination System permit regulations under 40 CFR part 122. These systems typically include individual drain systems, oil-water separators, air flotation units, equalization tanks, and biological treatment units.

Water seal controls means a seal pot, p-leg trap, or other type of trap filled with water that has a design capability to create a water barrier between the sewer line and the atmosphere.

[55 FR 8346, Mar. 7, 1990; 55 FR 12444, Apr. 3, 1990]

§ 61.342 Standards: General.

(a) An owner or operator of a facility at which the total annual benzene quantity from facility waste is less than 10 megagrams per year (Mg/yr) shall be exempt from the requirements of paragraphs (b) and (c) of this section. The total annual benzene quantity from facility waste is the sum of the annual benzene quantity for each waste streams at the facility that has a flow-weighted annual average water content greater than 10 percent.

The total annual benzene quantity from facility waste shall be determined in accordance with the procedures specified in § 61.355(a) of this subpart.

(b) Each owner or operator of a facility at which the total annual benzene quantity from facility waste is equal to or greater than 10 Mg/yr as determined in paragraph (a) of this section shall be in compliance with the requirements of paragraphs (c) through (g) of this section no later than March 7, 1992 or by the initial startup for a new source with an initial startup after this date.

(c) Each owner or operator of a facility at which the total annual benzene quantity from facility waste is equal to or greater than 10 Mg/yr as determined in paragraph (a) of this section shall manage and treat the facility waste as follows:

(1) For each waste stream, the owner or operator shall:

(i) Remove or destroy the benzene contained in the waste using a treatment process or wastewater treatment system that complies with the standards specified in § 61.348 of this subpart.

(ii) Comply with the standards specified in §§ 61.343 through 61.347 of this subpart for each waste management unit that receives or manages the waste stream prior to and during treatment of the waste stream in accordance with paragraph (c)(1)(i) of this section.

(iii) Each waste management unit used to manage or treat waste streams that will be recycled to a process shall comply with the standards specified in §§ 61.343 through 61.347 of this subpart. Once the waste stream is recycled to a process, the material is no longer subject to paragraph (c) of this section.

(2) A waste stream is exempt from paragraph (c)(1) of this section provided that the owner or operator demonstrates initially and, thereafter, at least once per year that the flow-weighted annual average benzene concentration for the waste stream is less than 10 ppmw as determined by the procedures specified in § 61.355(c) of this subpart.

(3) A process wastewater stream is exempt from paragraph (c)(1) of this section provided that the owner or operator demonstrates initially and, thereafter, at least once per year that one of the following conditions is met:

(i) The process wastewater stream flow rate is less than 0.02 liters per minute; or

(ii) The annual waste quantity of the process wastewater stream is less than 10 Mg/yr.

(d) As an alternative to the requirements specified in paragraph (c) of this section, an owner or operator of a facility at which the total annual benzene quantity from facility waste is equal to or greater than 10 Mg/yr as determined in paragraph (a) of this section may elect to manage and treat the facility waste as follows:

(1) The owner or operator shall manage and treat facility waste other than process wastewater in accordance with the requirements of paragraph (c)(1) of this section.

(2) The owner or operator shall manage and treat process wastewater in accordance with the following requirements:

(i) Process wastewater shall be treated to achieve a total annual benzene quantity from facility process wastewater less than 1 Mg/yr. Total annual benzene from facility process wastewater shall be determined by adding together the annual benzene quantity at the point of waste generation for each untreated process wastewater stream plus the annual benzene quantity exiting the treatment process for each process wastewater stream treated in accordance with the requirements of paragraph (c)(1)(i) of this section.

(ii) Each treated process wastewater stream identified in paragraph (d)(2)(i) of this section shall be managed and treated in accordance with paragraph (c)(1) of this section.

(iii) Each untreated process wastewater stream identified in paragraph (d)(2)(i) of this section is exempt from the requirements of paragraph (c)(1) of this section.

(e) Rather than treating the waste onsite, an owner or operator may elect to comply with paragraph (c)(1)(i) of this section by transferring the waste

offsite to another facility where the waste is treated in accordance with the requirements of paragraph (c)(1)(i) of this section. The owner or operator transferring the waste shall:

(1) Comply with the standards specified in §§ 61.343 through 61.347 of this subpart for each waste management unit that receives or manages the waste prior to shipment of the waste offsite.

(2) Include with each offsite waste shipment a notice stating that the waste contains benzene which is required to be managed and treated in accordance with the provisions of this subpart.

(f) Compliance with this subpart will be determined by review of facility records and results from tests and inspections using methods and procedures specified in § 61.355 of this subpart.

(g) Permission to use an alternative means of compliance to meet the requirements of §§ 61.342 through 61.352 of this subpart may be granted by the Administrator as provided in § 61.353 of this subpart. § 61.343 Standards: Tanks.

(a) Except as provided in § 61.351 of this subpart, the owner or operator shall meet the following standards for each tank in which the waste stream is placed in accordance with § 61.342(c)(1)(ii) of this subpart. The standards in this section apply to the treatment of the waste stream in a tank, including dewatering.

(1) The owner or operator shall install, operate, and maintain a fixed-roof and closed-vent system that routes all organic vapors vented from the tank to a control device.

(i) The fixed-roof shall meet the following requirements:

(A) The cover and all openings (e.g., access hatches, sampling ports, and gauge wells) shall be designed to operate with no detectable emissions as indicated by an instrument reading of less than 500 ppmv above background, as determined initially and thereafter at least once per year by the methods specified in § 61.355(h) of this subpart.

(B) Each opening shall be maintained in a closed, sealed position (e.g., covered by a lid that is gasketed and latched) at all times that waste is in

the tank except when it is necessary to use the opening for waste sampling or removal, or for equipment inspection, maintenance, or repair.

(ii) The closed-vent system and control device shall be designed and operated in accordance with the requirements of § 61.349 of this subpart.

(b) Each cover seal, access door, and all other openings shall be checked by visual inspection initially and quarterly thereafter to ensure that no cracks or gaps occur between the cover and tank wall and that access doors and other openings are closed and gasketed properly.

(c) Except as provided in § 61.350 of this subpart, when a broken seal or gasket or other problem is identified, or when detectable emissions are measured, first efforts at repair shall be made as soon as practicable, but not later than 45 calendar days after identification.

[55 FR 8346, Mar. 7, 1990, as amended at 55 FR 18331, May 2, 1990]

§ 61.344 Standards: Surface impoundments.

(a) The owner or operator shall meet the following standards for each surface impoundment in which waste is placed in accordance with § 61.342(c)(1)(ii) of this subpart:

(1) The owner or operator shall install, operate, and maintain on each surface impoundment a cover (e.g., air-supported structure or rigid cover) and closed-vent system that routes all organic vapors vented from the surface impoundment to a control device.

(i) The cover shall meet the following requirements:

(A) The cover and all openings (e.g., access hatches, sampling ports, and gauge wells) shall be designed to operate with no detectable emissions as indicated by an instrument reading of less than 500 ppmv above background, initially and thereafter at least once per year by the methods specified in § 61.355(h) of this subpart.

(B) Each opening shall be maintained in a closed, sealed position (e.g., covered by a lid that is gasketed and latched) at all times that waste is in the surface impoundment except when it is necessary to use the opening for waste sampling or removal, or

for equipment inspection, maintenance, or repair.

(C) The cover shall be used at all times that waste is placed in the surface impoundment except during removal of treatment residuals in accordance with 40 CFR 268.4 or closure of the surface impoundment in accordance with 40 CFR 264.228. (Note: the treatment residuals generated by these activities may be subject to the requirements of this part.)

(ii) The closed-vent system and control device shall be designed and operated in accordance with § 61.349 of this subpart.

(b) Each cover seal, access hatch, and all other openings shall be checked by visual inspection initially and quarterly thereafter to ensure that no cracks or gaps occur and that access hatches and other openings are closed and gasketed properly.

(c) Except as provided in § 61.350 of this subpart, when a broken seal or gasket or other problem is identified, or when detectable emissions are measured, first efforts at repair shall be made as soon as practicable, but not later than 15 calendar days after identification.

§ 61.345 Standards: Containers.

(a) The owner or operator shall meet the following standards for each container in which waste is placed in accordance with § 61.342(c)(1)(ii) of this subpart:

(1) The owner or operator shall install, operate, and maintain a cover on each container used to handle, transfer, or store waste in accordance with the following requirements:

(i) The cover and all openings (e.g., bungs, hatches, and sampling ports) shall be designed to operate with no detectable emissions as indicated by an instrument reading of less than 500 ppmv above background, initially and thereafter at least once per year by the methods specified in § 61.355(h) of this subpart.

(ii) Each opening shall be maintained in a closed, sealed position (e.g., covered by a lid that is gasketed and latched) at all times that waste is in the container except when it is neces-

sary to use the opening for waste loading, removal inspection, or sampling.

(2) Loading a pumpable waste into a container shall be performed by the owner or operator using a submerged fill pipe. The submerged fill pipe outlet shall extend to within two fill pipe diameters of the bottom of the container while the container is being loaded. During loading of the waste, the cover shall remain in place and all openings shall be maintained in a closed, sealed position except for those openings required for the submerged fill pipe and for venting of the container to prevent physical damage or permanent deformation of the container or cover.

(3) Treatment of a waste in a container, including aeration, thermal or other treatment, shall be performed by the owner or operator in a manner such that whenever it is necessary for the container to be open while the waste is being treated, the container is located under a cover (e.g., enclosure) with a closed-vent system that routes all organic vapors vented from the container to a control device.

(i) The cover and all openings (e.g., doors, hatches) shall be designed to operate with no detectable emissions as indicated by an instrument reading of less than 500 ppmv above background, initially and thereafter at least once per year by the methods specified in § 61.355(h) of this subpart.

(ii) The closed-vent system and control device shall be designed and operated in accordance with § 61.349 of this subpart.

(b) Each cover and all openings shall be visually inspected initially and quarterly thereafter to ensure that they are closed and gasketed properly.

(c) Except as provided in § 61.350 of this subpart, when a broken seal or gasket or other problem is identified, first efforts at repair shall be made as soon as practicable, but not later than 15 calendar days after identification.

§ 61.346 Standards: Individual drain systems.

(a) Except as provided in paragraph (b) of this section, the owner or operator shall meet the following standards for each individual drain system in

which waste is placed in accordance with § 61.342(c)(1)(ii) of this subpart:

(1) The owner or operator shall install, operate, and maintain on each drain system opening a cover and closed-vent system that routes all organic vapors vented from the drain system to a control device.

(i) The cover shall meet the following requirements:

(A) The cover and all openings (e.g., access hatches, sampling ports) shall be designed to operate with no detectable emissions as indicated by an instrument reading of less than 500 ppmv above background, initially and thereafter at least once per year by the methods specified in § 61.355(h) of this subpart.

(B) Each opening shall be maintained in a closed, sealed position (e.g., covered by a lid that is gasketed and latched) at all times that waste is in the drain system except when it is necessary to use the opening for waste sampling or removal, or for equipment inspection, maintenance, or repair.

(ii) The closed-vent system and control device shall be designed and operated in accordance with § 61.349 of this subpart.

(2) Each cover seal, access hatch, and all other openings shall be checked by visual inspection initially and quarterly thereafter to ensure that no cracks or gaps occur and that access hatches and other openings are closed and gasketed properly.

(3) Except as provided in § 61.350 of this subpart, when a broken seal or gasket or other problem is identified, or when detectable emissions are measured, first efforts at repair shall be made as soon as practicable, but not later than 15 calendar days after identification.

(b) As an alternative to complying with paragraph (a) of this section, an owner or operator may elect to comply with the following requirements:

(1) Each drain shall be equipped with water seal controls or a tightly sealed cap or plug.

(2) Each junction box shall be equipped with a cover and may have a vent pipe. The vent pipe shall be at least 90 cm (3 ft) in length and shall not exceed 10.2 cm (4 in) in diameter.

(i) Junction box covers shall have a tight seal around the edge and shall be kept in place at all times, except during inspection and maintenance.

(ii) One of the following methods shall be used to control emissions from the junction box vent pipe to the atmosphere:

(A) Equip the junction box with a system to prevent the flow of organic vapors from the junction box vent pipe to the atmosphere during normal operation. An example of such a system includes use of water seal controls. A flow indicator shall be installed, operated, and maintained on each junction box vent pipe to ensure that organic vapors are not vented from the junction box to the atmosphere during normal operation.

(B) Connect the junction box vent pipe to a closed-vent system and control device in accordance with § 61.349 of this subpart.

(3) Each sewer line shall not be open to the atmosphere and shall be covered or enclosed in a manner so as to have no visual gaps or cracks in joints, seals, or other emission interfaces.

(4) Equipment installed in accordance with paragraphs (b)(1), (b)(2), or (b)(3) of this section shall be inspected as follows:

(i) Each drain using water seal controls shall be checked by visual or physical inspection initially and thereafter quarterly for indications of low water levels or other conditions that would reduce the effectiveness of water seal controls.

(ii) Each drain using a tightly sealed cap or plug shall be visually inspected initially and thereafter quarterly to ensure caps or plugs are in place and properly installed.

(iii) Each junction box shall be visually inspected initially and thereafter quarterly to ensure that the cover is in place and to ensure that the cover has a tight seal around the edge.

(iv) The unburied portion of each sewer line shall be visually inspected initially and thereafter quarterly for indication of cracks, gaps, or other problems that could result in benzene emissions.

(5) Except as provided in § 61.350 of this subpart, when a broken seal, gap, crack or other problem is identified,

first efforts at repair shall be made as soon as practicable, but not later than 15 calendar days after identification.

§ 61.347 Standards: Oil-water separators.

(a) Except as provided in § 61.352 of this subpart, the owner or operator shall meet the following standards for each oil-water separator in which waste is placed in accordance with § 61.342(c)(1)(ii) of this subpart:

(1) The owner or operator shall install, operate, and maintain a fixed-roof and closed-vent system that routes all organic vapors vented from the oil-water separator to a control device.

(i) The fixed-roof shall meet the following requirements:

(A) The cover and all openings (e.g., access hatches, sampling po. and gauge wells) shall be designed to operate with no detectable emissions as indicated by an instrument reading of less than 500 ppmv above background, as determined initially and thereafter at least once per year by the methods specified in § 61.355(h) of this subpart.

(B) Each opening shall be maintained in a closed, sealed position (e.g., covered by a lid that is gasketed and latched) at all times that waste is in the oil-water separator except when it is necessary to use the opening for waste sampling or removal, or for equipment inspection, maintenance, or repair.

(ii) The closed-vent system and control device shall be designed and operated in accordance with the requirements of § 61.349 of this subpart.

(b) Each cover seal, access hatch, and all other openings shall be checked by visual inspection initially and quarterly thereafter to ensure that no cracks or gaps occur between the cover and oil-water separator wall and that access hatches and other openings are closed and gasketed properly.

(c) Except as provided in § 61.350 of this subpart, when a broken seal or gasket or other problem is identified, or when detectable emissions are measured, first efforts at repair shall be made as soon as practicable, but not later than 15 calendar days after identification.

§ 61.348 Standards: Treatment processes.

(a) Except as provided in paragraph (a)(5) of this section, the owner or operator shall treat the waste stream in accordance with the following requirements:

(1) The owner or operator shall design, install, operate, and maintain a treatment process that either:

(i) Removes benzene from the waste stream to a level less than 10 parts per million by weight (ppmw) on a flow-weighted annual average basis,

(ii) Removes benzene from the waste stream by 99 percent or more on a mass basis, or

(iii) Destroys benzene in the waste stream by incinerating the waste in a combustion unit that achieves a destruction efficiency of 99 percent or greater for benzene.

(2) Each treatment process complying with paragraphs (a)(1)(i) or (a)(1)(ii) of this section shall be designed and operated in accordance with the appropriate waste management unit standards specified in §§ 61.343 through 61.347 of this subpart. For example, if a treatment process is a tank, then the owner or operator shall comply with § 61.343 of this subpart.

(3) For the purpose of complying with the requirements specified in paragraph (a)(1)(i) of this section, the intentional or unintentional reduction in the benzene concentration of a waste stream by dilution of the waste stream with other wastes or materials is not allowed.

(4) An owner or operator may aggregate or mix together individual waste streams to create a combined waste stream for the purpose of facilitating treatment of waste to comply with the requirements of paragraph (a)(1) of this section except as provided in paragraph (a)(5) of this section.

(5) If an owner or operator aggregates or mixes any combination of process wastewater, product tank drawdown, or landfill leachate subject to § 61.342(c)(1) of this subpart together with other waste streams to create a combined waste stream for the purpose of facilitating management or treatment of waste in a wastewater treatment system, then the wastewater treatment system shall be

operated in accordance with paragraph (b) of this section.

(b) The owner or operator that aggregates or mixes individual waste streams as defined in paragraph (a)(5) of this section for management and treatment in a wastewater treatment system shall comply with the following requirements:

(1) The owner or operator shall design and operate each waste management unit that comprises the wastewater treatment system in accordance with the appropriate standards specified in §§ 61.343 through 61.347 of this subpart.

(2) The provisions of paragraph (b)(1) of this section do not apply to any waste management unit that the owner or operator demonstrates to meet the following conditions initially and, thereafter, at least once per year:

(i) The benzene content of each waste stream entering the waste management unit is less than 10 ppmw on a flow-weighted annual average basis as determined by the procedures specified in § 61.355(c) of this subpart; and

(ii) The total annual benzene quantity contained in all waste streams managed or treated in exempt waste management units comprising the facility wastewater treatment systems is less than 1 Mg/yr. For this determination, total annual benzene quantity shall be calculated as follows:

(A) The total annual benzene quantity shall be calculated as the sum of the individual benzene quantities determined at each location where a waste stream first enters an exempt waste management unit. The benzene quantity discharged from an exempt waste management unit shall not be included in this calculation.

(B) The annual benzene quantity in a waste stream managed or treated in an enhanced biodegradation unit shall not be included in the calculation of the total annual benzene quantity, if the enhanced biodegradation unit is the first exempt unit in which the waste is managed or treated. A unit shall be considered enhanced biodegradation provided that the process generates biomass, some of which is recycled as well as periodically removed from the unit; and typically operates at a food-to-microorganism

ratio in the range of 0.05 to 1.0 kg of biological oxygen demand per kg of biomass per day, a mixed liquor suspended solids ratio in the range of 1 to 8 grams per liter, and a residence time in the range of 3 to 36 hours.

(c) The owner and operator shall demonstrate that each treatment process or wastewater treatment system unit, except as provided in paragraph (d) of this section, achieves the appropriate conditions specified in paragraphs (a) or (b) of this section in accordance with the following requirements:

(1) Engineering calculations in accordance with requirements specified in § 61.356(e) of this subpart; or

(2) Performance tests conducted using the test methods and procedures that meet the requirements specified in § 61.355 of this subpart.

(d) A treatment process or waste stream is in compliance with the requirements of this subpart and exempt from the requirements of paragraph (c) of this section provided that the owner or operator documents that the treatment process or waste stream is in compliance with other regulatory requirements as follows:

(1) The treatment process is a hazardous waste incinerator for which the owner or operator has been issued a final permit under 40 CFR part 270 and complies with the requirements of 40 CFR part 264, subpart 0;

(2) The treatment process is an industrial furnace or boiler burning hazardous waste for energy recovery for which the owner or operator has been issued a final permit under 40 CFR part 270 and complies with the requirements of 40 CFR part 266, subpart D;

(3) The waste stream is treated by a means or to a level that meets benzene-specific treatment standards in accordance with the Land Disposal Restrictions under 40 CFR part 268, and the treatment process is designed and operated with a closed-vent system and control device meeting the requirements of § 61.349 of this subpart;

(4) The waste stream is treated by a means or to a level that meets benzene-specific effluent limitations or performance standards in accordance

with the Effluent Guidelines and Standards under 40 CFR parts 401-464, and the treatment process is designed and operated with a closed-vent system and control device meeting the requirements of § 61.349 of this subpart; or

(5) The waste stream is discharged to an underground injection well for which the owner or operator has been issued a final permit under 40 CFR part 270 and complies with the requirements of 40 CFR part 122.

(e) If the treatment process or wastewater treatment system unit has any openings (e.g., access doors, hatches, etc.), all such openings shall be sealed (e.g., gasketed, latched, etc.) and kept closed at all times when waste is being treated, except during inspection and maintenance.

(f) Each seal, access door, and all other openings shall be checked by visual inspections initially and quarterly thereafter to ensure that no cracks or gaps occur and that openings are closed and gasketed properly.

(g) Except as provided in § 61.350 of this subpart, when a broken seal or gasket or other problem is identified, first efforts at repair shall be made as soon as practicable, but not later than 15 calendar days after identification.

(h) Except for treatment processes complying with paragraph (d) of this section, the Administrator may request at any time an owner or operator demonstrate that a treatment process or wastewater treatment system unit meets the applicable requirements specified in paragraphs (a) or (b) of this section by conducting a performance test using the test methods and procedures as required in § 61.355 of this subpart.

(i) The owner or operator of a treatment process or wastewater treatment system unit that is used to comply with the provisions of this section shall monitor the unit in accordance with the applicable requirements in § 61.354 of this subpart.

§ 61.349 Standards: Closed-vent systems and control devices.

(a) For each closed-vent system and control device used to comply with standards in accordance with §§ 61.343

through 61.348 of this subpart, the owner or operator shall properly design, install, operate, and maintain the closed-vent system and control device in accordance with the following requirements:

(1) The closed-vent system shall:

(i) Be designed to operate with no detectable emissions as indicated by an instrument reading of less than 500 ppmv above background, as determined initially and thereafter at least once per year by the methods specified in § 61.355(h) of this subpart.

(ii) A flow indicator shall be installed on each vent stream to the control device to ensure that the vapors are being routed to the device. The flow indicator shall be installed in the vent stream at the nearest feasible point to the control device inlet but before being combined with other vent streams.

(iii) All gauging and sampling devices shall be gas-tight except when gauging or sampling is taking place.

(2) The control device shall be designed and operated in accordance with the following conditions:

(i) An enclosed combustion device (e.g., a vapor incinerator, boiler, or process heater) shall meet one of the following conditions:

(A) Reduce the organic emissions vented to it by 95 weight percent or greater;

(B) Achieve a total organic compound concentration of 20 ppmv on a dry basis corrected to 3 percent oxygen; or

(C) Provide a minimum residence time of 0.5 seconds at a minimum temperature of 760°C. If a boiler or process heater issued as the control device, then the vent stream shall be introduced into the flame zone of the boiler or process heater.

(ii) A vapor recovery system (e.g., carbon absorption system or condenser) shall recover the organic emissions vented to it with an efficiency of 95 weight percent or greater.

(iii) A flare shall comply with the requirements of 40 CFR 60.18.

(b) Each closed-vent system and control device used to comply with this subpart shall be operated at all times when waste is placed in the waste management unit vented to the con-

trol device except when maintenance or repair of the waste management unit cannot be completed without a shutdown of the control device.

(c) An owner and operator shall demonstrate that each control device, except for a flare, achieves the appropriate conditions specified in paragraph (a)(2) of this section by using one of the following methods:

(i) Engineering calculations in accordance with requirements specified in § 61.356(f) of this subpart; or

(ii) Performance tests conducted using the test methods and procedures that meet the requirements specified in § 61.355 of this subpart.

(d) An owner or operator shall demonstrate compliance of each flare in accordance with paragraph (a)(2)(iii) of this section.

(e) The Administrator may request at any time an owner or operator demonstrate that a control device meets the applicable conditions specified in paragraph (a)(2) of this section by conducting a performance test using the test methods and procedures as required in § 61.355 of this subpart.

(f) Each closed-vent system and control device shall be visually inspected initially and quarterly thereafter. The visual inspection shall include inspection of ductwork and piping and connections to covers and control devices for evidence of visible defects such as holes in ductwork or piping and loose connections.

(g) Except as provided in § 61.350 of this subpart, if visible defects are observed during an inspection, or if other problems are identified, or if detectable emissions are measured, a first effort to repair the closed-vent system and control device shall be made as soon as practicable but no later than 5 calendar days after detection. Repair shall be completed no later than 15 calendar days after the emissions are detected or the visible defect is observed.

(h) The owner or operator of a control device that is used to comply with the provisions of this section shall monitor the control device in accordance with § 61.354(c) of this subpart.

[55 FR 8346, Mar. 7, 1990; 55 FR 12444, Apr. 3, 1990]

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§ 61.350 Standards: Delay of repair.

(a) Delay of repair of facilities or units that are subject to the provisions of this subpart will be allowed if the repair is technically impossible without a complete or partial facility or unit shutdown.

(b) Repair of such equipment shall occur before the end of the next facility or unit shutdown.

§ 61.351 Alternative standards for tanks.

(a) As an alternative to the standards for tanks specified in § 61.343 of this subpart, an owner or operator may elect to comply with one of the following:

(1) A fixed roof and internal floating roof meeting the requirements in 40 CFR 60.112b(a)(1);

(2) An external floating roof meeting the requirements of 40 CFR 60.112(a)(2); or

(3) An alternative means of emission limitation as described in 40 CFR 60.114b.

(b) If an owner or operator elects to comply with the provisions of this section, then the owner or operator is exempt from the provisions of § 61.343 of this subpart applicable to the same facilities.

§ 61.352 Alternative standards for oil-water separators.

(a) As an alternative to the standards for oil-water separators specified in § 61.347 of this subpart, an owner or operator may elect to comply with one of the following:

(1) A floating roof meeting the requirements in 40 CFR 60.693-2(a); or

(2) An alternative means of emission limitation as described in 40 CFR 60.594.

(b) For portions of the oil-water separator where it is infeasible to construct and operate a floating roof, such as over the weir mechanism, a fixed roof vented to a vapor control device that meets the requirements in §§ 61.347 and 61.349 of this subpart shall be installed and operated.

(c) Except as provided in paragraph (b) of this section, if an owner or operator elects to comply with the provisions of this section, then the owner or operator is exempt from the provisions

in § 61.347 of this subpart applicable to the same facilities.

§ 61.353 Alternative means of emission limitation.

(a) If, in the Administrator's judgment, an alternative means of emission limitation will achieve a reduction in benzene emissions at least equivalent to the reduction in benzene emissions achieved by the applicable requirements in §§ 61.342 through 61.349 of this subpart, the Administrator will publish in the *FEDERAL REGISTER* a notice permitting the use of the alternative means for purposes of compliance with that requirement. The notice may condition the permission on requirements related to the operation and maintenance of the alternative means.

(b) Any notice under paragraph (a) of this section shall be published only after public notice and an opportunity for a hearing.

(c) Any person seeking permission under this section shall collect, verify, and submit to the Administrator information showing that the alternative means achieves equivalent emission reductions.

§ 61.354 Monitoring of operations.

(a) Except for a treatment process or waste stream complying with § 61.348(d), the owner or operator shall monitor each treatment process or wastewater treatment system unit to ensure the unit is properly operated and maintained by one of the following monitoring procedures:

(1) Measure the benzene concentration of the waste stream exiting the treatment process complying with paragraph (a)(1)(i) of this section or the wastewater stream exiting the wastewater treatment unit complying with paragraph (b) of this section at least once per month by collecting and analyzing one or more samples using the procedures specified in § 61.355(c)(2) of this subpart.

(2) Install, calibrate, operate, and maintain according to manufacturer's specifications equipment to continuously monitor and record a process parameter (or parameters) for the treatment process or wastewater treatment

system unit that indicates proper system operation. The owner or operator shall inspect at least once each operating day the data recorded by the monitoring equipment (e.g., temperature monitor or flow indicator) to ensure that the unit is operating properly.

(b) If an owner or operator complies with the requirements of § 61.348(b) of this subpart, then the owner or operator shall install, calibrate, operate, and maintain according to manufacturer's specifications equipment to continuously monitor and record the flow rate of each wastewater stream exiting the wastewater treatment system.

(c) An owner or operator subject to the requirements in § 61.349 of this subpart shall install, calibrate, maintain, and operate according to the manufacturer's specifications a device to continuously monitor the control device operation as specified in the following paragraphs, unless alternative monitoring procedures or requirements are approved for that facility by the Administrator. The owner or operator shall inspect at least once each operating day the data recorded by the monitoring equipment (e.g., temperature monitor or flow indicator) to ensure that the control device is operating properly.

(1) For a thermal vapor incinerator, a temperature monitoring device equipped with a continuous recorder. The device shall have an accuracy of ± 1 percent of the temperature being monitored in $^{\circ}\text{C}$ or $\pm 0.5^{\circ}\text{C}$, whichever is greater. The temperature sensor shall be installed at a representative location in the combustion chamber.

(2) For a catalytic vapor incinerator, a temperature monitoring device equipped with a continuous recorder. The device shall be capable of monitoring temperature at two locations, and have an accuracy of ± 1 percent of the temperature being monitored in $^{\circ}\text{C}$ or $\pm 0.5^{\circ}\text{C}$, whichever is greater. One temperature sensor shall be installed in the vent stream at the nearest feasible point to the catalyst bed inlet and a second temperature sensor shall be installed in the vent stream at the nearest feasible point to the catalyst bed outlet.

(3) For a flare, a monitoring device in accordance with 40 CFR 60.18(f)(2) equipped with a continuous recorder.

(4) For a boiler or process heater having a design heat input capacity less than 44 megawatts (MW), a temperature monitoring device equipped with a continuous recorder. The device shall have an accuracy of ± 1 percent of the temperature being monitored in $^{\circ}\text{C}$ or $\pm 0.5^{\circ}\text{C}$, whichever is greater. The temperature sensor shall be installed at a representative location in the combustion chamber.

(5) For a boiler or process heater having a design heat input capacity greater than or equal to 44 MW, a monitoring device equipped with a continuous recorder to measure a parameter(s) that indicates good combustion operating practices are being used.

(6) For a condenser, either: (i) A monitoring device equipped with a continuous recorder to measure the concentration level of the organic compounds in the exhaust vent stream from the condenser; or (ii) A temperature monitoring device equipped with a continuous recorder. The device shall be capable of monitoring temperature at two locations, and have an accuracy of ± 1 percent of the temperature being monitored in $^{\circ}\text{C}$ or $\pm 0.5^{\circ}\text{C}$, whichever is greater. One temperature sensor shall be installed at a location in the exhaust stream from the condenser, and a second temperature sensor shall be installed at a location in the coolant fluid exiting the condenser.

(7) For a carbon adsorption system that regenerates the carbon bed directly in the control device such as a fixed-bed carbon adsorber, either:

(i) A monitoring device equipped with a continuous recorder to measure the concentration level of the organic compounds in the exhaust vent stream from the carbon bed; or (ii) A monitoring device equipped with a continuous recorder to measure a parameter that indicates the carbon bed is regenerated on a regular, predetermined time cycle.

(8) For a vapor recovery system other than a condenser or carbon adsorption system, a monitoring device equipped with a continuous recorder

to measure the concentration level of the organic compounds in the exhaust vent stream from the control device.

(d) For a carbon adsorption system that does not regenerate the carbon bed directly on site in the control device (e.g., a carbon canister), the concentration level of the organic compounds in the exhaust vent stream from the carbon adsorption system shall be monitored on a regular schedule, and the existing carbon shall be replaced with fresh carbon immediately when carbon breakthrough is indicated. The device shall be monitored on a daily basis or at intervals no greater than 20 percent of the design carbon replacement interval, whichever is greater. As an alternative to conducting this monitoring, an owner or operator may replace the carbon in the carbon adsorption system with fresh carbon at a regular predetermined time interval that is less than the carbon replacement interval that is determined by the maximum design flow rate and organic concentration in the gas stream vented to the carbon adsorption system.

(e) An alternative operation or process parameter may be monitored if it can be demonstrated that another parameter will ensure that the control device is operated in conformance with these standards and the control device's design specifications.

§ 61.355 Test methods, procedures, and compliance provisions.

(a) An owner or operator shall determine the total annual benzene quantity from facility waste by the following procedure:

(1) For each waste stream subject to this subpart having a flow-weighted annual average water content greater than 10 percent water, the owner or operator shall:

(i) Determine the annual waste quantity for each waste stream at the point of waste generation using the procedures specified in paragraph (b) of this section.

(ii) Determine the flow-weighted annual average benzene concentration for each waste stream at the point of waste generation using the procedures specified in paragraph (c) of this section.

(iii) Calculate the annual benzene quantity for each waste stream by multiplying the annual waste quantity of the waste stream times the flow-weighted annual average benzene concentration.

(2) Total annual benzene quantity from facility waste is calculated by adding together the annual benzene quantity for each waste stream.

(3) If the total annual benzene quantity from facility waste is equal to or greater than 10 Mg/yr, then the owner or operator shall comply with the requirements of § 61.342(c) or (d) of this subpart.

(4) If the total annual benzene quantity from facility waste is less than 10 Mg/yr but is equal to or greater than 1 Mg/yr, then the owner or operator shall:

(i) Comply with the recordkeeping requirements of § 61.356 and reporting requirements of § 61.357 of this subpart; and

(ii) Repeat the determination of total annual benzene quantity from facility waste at least once per year and whenever there is a change in the process generating the waste that could cause the total annual benzene quantity from facility waste to increase to 10 Mg/yr or more.

(5) If the total annual benzene quantity from facility waste is less than 1 Mg/yr, then the owner or operator shall:

(i) Comply with the recordkeeping requirements of § 61.356 and reporting requirements of § 61.357 of this subpart; and

(ii) Repeat the determination of total annual benzene quantity from facility waste whenever there is a change in the process generating the waste that could cause the total annual benzene quantity from facility waste to increase to 1 Mg/yr or more.

(b) An owner or operator shall determine the annual waste quantity for each waste stream by one of the following methods:

(1) Selecting the highest annual quantity of waste managed from historical records representing the most recent 5 years of operation or, if the facility has been in service for less than 5 years but at least 1 year, from

historical records representing the total operating life of the facility;

(2) Using the maximum design capacity of the waste management unit; or

(3) Measurements that are representative of maximum waste generation rates.

(c) An owner or operator shall determine the flow-weighted annual average benzene concentration for each waste stream by one of the following methods:

(1) *Knowledge of the waste.* The owner or operator shall provide sufficient information to document the flow-weighted annual average benzene concentration of each waste stream. Examples of information that could constitute knowledge include material balances, records of chemicals purchases, or previous test results provided the results are still relevant to the current waste stream conditions. If test data are used, then the owner or operator shall provide documentation describing the testing protocol and the means by which sampling variability and analytical variability were accounted for in the determination of the flow-weighted annual average benzene concentration for the waste stream.

(2) Measurements of the benzene concentration in the waste stream in accordance with the following procedures:

(i) Collect a minimum of three representative samples from each waste stream. Where feasible, samples shall be taken from an enclosed pipe prior to the waste being exposed to the atmosphere.

(ii) For waste in enclosed pipes, the following procedures shall be used:

(A) Samples shall be collected prior to the waste being exposed to the atmosphere in order to minimize the loss of benzene prior to sampling.

(B) A static mixer shall be installed in the process line or in a by-pass line unless the owner or operator demonstrates that installation of a static mixer in the line is not necessary to accurately determine the benzene concentration of the waste stream.

(C) The sampling tap shall be located within two pipe diameters of the static mixer outlet.

(D) Prior to the initiation of sampling, sample lines and cooling coil shall be purged with at least four volumes of waste.

(E) After purging, the sample flow shall be directed to a sample container and the tip of the sampling tube shall be kept below the surface of the waste during sampling to minimize contact with the atmosphere.

(F) Samples shall be collected at a flow rate such that the cooling coil is able to maintain a waste temperature less than 10°C.

(G) After filling, the sample container shall be capped immediately (within 5 seconds) to leave a minimum headspace in the container.

(H) The sample containers shall immediately be cooled and maintained at a temperature below 10°C for transfer to the laboratory.

(iii) When sampling from an enclosed pipe is not feasible, a minimum of three representative samples shall be collected in a manner to minimize exposure of the sample to the atmosphere and loss of benzene prior to sampling.

(iv) Each waste sample shall be analyzed using one of the following test methods for determining the benzene concentration in a waste stream:

(A) Method 8020, Aromatic Volatile Organics, in "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods," EPA Publication No. SW-846 (incorporation by reference as specified in § 61.18 of this part);

(B) Method 8021, Volatile Organic Compounds in Water by Purge and Trap Capillary Column Gas Chromatography with Photoionization and Electrolytic Conductivity Detectors in Series in "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods," EPA Publication No. SW-846 (incorporation by reference as specified in § 61.18 of this part);

(C) Method 8240, Gas Chromatography/Mass Spectrometry for Volatile Organics in "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods," EPA Publication No. SW-846 (incorporation by reference as specified in § 61.18 of this part);

(D) Method 8260, Gas Chromatography/Mass Spectrometry for Volatile Organics: Capillary Column Tech-

nique in "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods," EPA Publication No. SW-846 (incorporation by reference as specified in § 61.18 of this part);

(E) Method 602, Purgeable Aromatics, as described in 40 CFR part 136, appendix A, Test Procedures for Analysis of Organic Pollutants, for wastewaters for which this is an approved EPA method; or

(F) Method 624, Purgeables, as described in 40 CFR part 136, appendix A, Test Procedures for Analysis of Organic Pollutants, for wastewaters for which this is an approved EPA method.

(v) The flow-weighted annual average benzene concentration shall be calculated by averaging the results of the sample analyses as follows:

$$\bar{C} = \frac{1}{n} \times \sum_{i=1}^n (Q_i)(C_i)$$

Where:

$\bar{C}$ = Flow-weighted annual average benzene concentration for waste stream, ppmw.

Q_i = Total annual waste quantity for waste stream, kg/yr.

n = Number of waste samples (at least 3).

Q_i = Annual waste quantity for waste stream represented by C_i , kg/yr.

C_i = Measured concentration of benzene in waste sample i , ppmw.

(d) An owner or operator using performance tests to demonstrate compliance of a treatment process with § 61.348(a)(1)(i) of this subpart shall measure the flow-weighted annual average benzene concentration of the waste stream exiting the treatment process by collecting and analyzing a minimum of three representative samples of the waste stream using the procedure in paragraph (c)(2) of this section. The test shall be conducted under conditions that exist when the treatment process is operating at the

highest inlet waste stream flow rate and benzene content expected to occur. Operations during periods of startup, shutdown, and malfunction shall not constitute representative conditions for the purpose of a test. The owner or operator shall record all process information as is necessary to document the operating conditions during the test.

(e) An owner or operator using performance tests to demonstrate compliance of a treatment process with § 61.348(a)(1)(ii) of this subpart shall determine the percent reduction of benzene in the waste stream on a mass basis by the following procedure:

(1) The test shall be conducted under conditions that exist when the treatment process is operating at the highest inlet waste stream flow rate and benzene content expected to occur. Operations during periods of startup, shutdown, and malfunction shall not constitute representative conditions for the purpose of a test. The owner or operator shall record all process information as is necessary to document the operating conditions during the test.

(2) All testing equipment shall be prepared and installed as specified in the appropriate test methods.

(3) The mass flow rate of benzene entering the treatment process (E_b) shall be determined by computing the product of the flow rate of the waste stream entering the treatment process, as determined by the inlet flow meter, and the benzene concentration of the waste stream, as determined using the sampling and analytical procedures specified in paragraph (c) of this section. Three grab samples of the waste shall be taken at equally spaced time intervals over a 1-hour period. Each 1-hour period constitutes a run, and the performance test shall consist of a minimum of 3 runs conducted over a 3-hour period. The mass flow rate of benzene entering the treatment process is calculated as follows:

$$E_b = \frac{K}{n \times 10^6} \left(\sum_{i=1}^n V_i C_i \right)$$

Where:

E_b = Mass flow rate of benzene entering the treatment process, kg/hour.

K = Density of the waste stream, kg/m³.

V_i = Average volume flow rate of waste entering the treatment process during each run i , m³/hour.

C_i = Average concentration of benzene in the waste stream entering the treatment process during each run i , ppmw.

n = Number of runs.

(4) The mass flow rate of benzene exiting the treatment process (E_a) shall be determined by computing the product of the flow rate of the waste stream exiting the treatment process, as determined by the inlet flow meter,

$$E_a = \frac{K}{n \times 10^6} \left(\sum_{i=1}^n V_i C_i \right)$$

Where:

E_a = Mass flow rate of benzene exiting the treatment process, kg/hour.

K = Density of the waste stream, kg/m³.

V_i = Average volume flow rate of waste exiting the treatment process during each run i , m³/hour.

C_i = Average concentration of benzene in the waste stream exiting the treatment process during each run i , ppmw.

n = Number of runs.

(5) The percent reduction across the treatment process shall be calculated as follows:

$$R = \frac{E_b - E_a}{E_b} \times 100$$

Where:

R = Control efficiency of the treatment process, percent.

E_b = Mass flow rate of benzene entering the treatment process, kg/hour.

E_a = Mass flow rate of benzene exiting the treatment process, kg/hour.

(f) An owner or operator using performance tests to demonstrate compliance of a treatment process with § 61.348(a)(1)(iii) of this subpart shall determine the benzene destruction efficiency for the combustion unit by the following procedure:

and the benzene concentration of the waste stream, as determined using the sampling and analytical procedures specified in paragraph (c) of this section. Three grab samples of the waste shall be taken at equally spaced time intervals over a 1-hour period. Each 1-hour period constitutes a run, and the performance test shall consist of a minimum of 3 runs conducted over the same 3-hour period at which the mass flow rate of benzene entering the treatment process is determined. The mass flow rate of benzene exiting the treatment process is calculated as follows:

(1) The test shall be conducted under conditions that exist when the combustion unit is operating at the highest inlet waste stream flow rate and benzene content expected to occur. Operations during periods of startup, shutdown, and malfunction shall not constitute representative conditions for the purpose of a test. The owner or operator shall record all process information necessary to document the operating conditions during the test.

(2) All testing equipment shall be prepared and installed as specified in the appropriate test methods.

(3) The mass flow rate of benzene entering the combustion unit shall be determined by computing the product of the flow rate of the waste stream entering the combustion unit, as determined by the inlet flow meter, and the benzene concentration of the waste stream, as determined using the sampling procedures in paragraph (c) of this section. Three grab samples of the waste shall be taken at equally spaced time intervals over a 1-hour period. Each 1-hour period constitutes a run, and the performance test shall consist of a minimum of 3 runs conducted over a 3-hour period. The mass flow rate of benzene into the combustion unit is calculated as follows:

$$E_a = \frac{K}{n \times 10^6} \left(\sum_{i=1}^n V_i C_i \right)$$

Where:

E_a = Mass flow rate of benzene into the combustion unit, kg/hour.

K = Density of the waste stream, kg/m³.

V_i = Average volume flow rate of waste entering the combustion unit during each run i , m³/hour.

C_i = Average concentration of benzene in the waste stream entering the combustion unit during each run i , ppmw.

n = Number of runs.

(4) The mass flow rate of benzene exiting the combustion unit exhaust stack shall be determined as follows:

(i) The time period for the test shall not be less than 3 hours during which at least 3 stack gas samples are collected and be the same time period at which the mass flow rate of benzene entering the treatment process is determined. Each sample shall be collected over a 1-hour period (e.g., in a tedlar bag) to represent a time-integrated composite sample and each 1-hour period shall correspond to the periods when the waste feed is sampled.

(ii) A run shall consist of a 1-hour period during the test. For each run:

(A) The reading from each measurement shall be recorded;

(B) The volume exhausted shall be determined using method 2, 2A, 2C, or 2D from appendix A of 40 CFR part 60, as appropriate.

(C) The average benzene concentration in the exhaust downstream of the combustion unit shall be determined using method 18 from appendix A of 40 CFR part 60.

(iii) The mass of benzene emitted during each run shall be calculated as follows:

$$M_i = KVC(10^{-6})$$

Where:

M_i = Mass of benzene emitted during run i , kg.

V = Volume of air-vapor mixture exhausted at standard conditions, m³.

C = Concentration of benzene measured in the exhaust, ppmv.

K = Conversion factor = 3.24 kg/m³ for benzene.

(iv) The benzene mass emission rate in the exhaust shall be calculated as follows:

$$E_a = \left(\sum_{i=1}^n M_i \right) / T$$

Where:

E_a = Mass flow rate of benzene emitted, kg/hour.

M_i = Mass of benzene emitted during run i , kg.

T = Total time of all runs, hour.

n = Number of runs.

(5) The benzene destruction efficiency for the combustion unit shall be calculated as follows:

$$R = \frac{E_b - E_a}{E_b} \times 100$$

Where:

R = Benzene destruction efficiency for the combustion unit, percent.

E_b = Mass flow rate of benzene into the combustion unit, kg/hour.

E_a = Mass flow of benzene from the combustion unit, kg/hour.

(g) An owner or operator using performance tests to demonstrate compliance of a wastewater treatment system unit with § 61.348(b)(1) of this subpart shall measure the flow-weighted annual average benzene concentration of the wastewater stream exiting the unit by collecting and analyzing a minimum of three representative samples of the waste stream using the procedures in paragraph (c)(2) of this section. The test shall be conducted under conditions that exist when the wastewater treatment system is operating at the highest inlet wastewater stream flow rate and benzene content expected to occur. Operations during

periods of startup, shutdown, and malfunction shall not constitute representative conditions for the purpose of a test. The owner or operator shall record all process information as is necessary to document the operating conditions during the test.

(h) An owner or operator shall test equipment for compliance with no detectable emissions as required in §§ 61.343 through 61.347, and § 61.349 of this subpart in accordance with the following requirements:

(1) Monitoring shall comply with method 21 from appendix A of 40 CFR part 60.

(2) The detection instrument shall meet the performance criteria of method 21.

(3) The instrument shall be calibrated before use on each day of its use by the procedures specified in method 21.

(4) Calibration gases shall be:

(i) Zero air (less than 10 ppm of hydrocarbon in air); and

(ii) A mixture of methane or n-hexane and air at a concentration of approximately, but less than, 10,000 ppm methane or n-hexane.

(5) The background level shall be determined as set forth in method 21.

(6) The instrument probe shall be traversed around all potential leak interfaces as close as possible to the interface as described in method 21.

(7) The arithmetic difference between the maximum concentration indicated by the instrument and the background level is compared to 500 ppm for determining compliance.

(i) An owner or operator using a performance test to demonstrate compliance of a control device with the organic reduction efficiency requirement specified under § 61.349(a)(2) of this subpart shall use the following procedures:

(1) The test shall be conducted under conditions that exist when the waste management unit vented to the control device is operating at the highest load or capacity level expected to occur. Operations during periods of startup, shutdown, and malfunction shall not constitute representative conditions for the purpose of a test. The owner or operator shall record all process information necessary to docu-

ment the operating conditions during the test.

(2) Sampling sites shall be selected using method 1 or 1A from appendix A of 40 CFR part 60, as appropriate.

(3) The mass flow rate of organics entering and exiting the control device shall be determined as follows:

(i) The time period for the test shall not be less than 3 hours during which at least 3 stack gas samples are collected. Samples of the vent stream entering and exiting the control device shall be collected during the same time period. Each sample shall be collected over a 1-hour period (e.g., in a tedlar bag) to represent a time-integrated composite sample.

(ii) A run shall consist of a 1-hour period during the test. For each run:

(A) The reading from each measurement shall be recorded;

(B) The volume exhausted shall be determined using method 2, 2A, 2C, or 2D from appendix A of 40 CFR part 60, as appropriate;

(C) The organic concentration in the vent stream entering and exiting the control shall be determined using method 18 from appendix A of 40 CFR part 60.

(iii) The mass of organics entering and exiting the control device during each run shall be calculated as follows:

$$M_{ei} = K \sum_{i=1}^n C_{ei} MW_i (10^{-9})$$

$$M_{exj} = K \sum_{j=1}^n C_{exj} MW_j (10^{-9})$$

Where:

M_{ei} = Mass of organics in the vent stream entering the control device during run i, kg.

M_{exj} = Mass of organics in vent stream exiting the control device during run j, kg.

V_{ei} = Volume of vent stream entering the control device during run i at standard conditions, m³.

V_{exj} = Volume of vent stream exiting the control device during run j at standard conditions, m³.

C_{ai} = Organic concentration of compound i measured in the vent stream entering the control device as determined by Method 18, ppm by volume on a dry basis.

C_{vi} = Organic concentration of compound i measured in the vent stream exiting the control device as determined by method 18, ppm by volume on a dry basis.

MW_i = Molecular weight of organic compound i in the vent stream kg/kg-mol.

n = Number of organic compounds in the vent stream.

K = Conversion factor for molar volume = $0.0416 \text{ kg-mol/m}^3$ (at 293°K and 760 mm Hg).

10^{-6} = Conversion from ppm, ppm^{-1} .

(iv) The mass flow rate of organics entering and exiting the control device shall be calculated as follows:

$$E_a = \left(\sum_{j=1}^n M_{aj} \right) / T$$

$$E_v = \left(\sum_{j=1}^n M_{vj} \right) / T$$

Where:

E_a = Mass flow rate of organics entering the control device, kg/hour.

E_v = Mass flow rate of organics exiting the control device, kg/hour.

M_{aj} = Mass of organics in the vent stream entering the control device during run j , kg.

M_{vj} = Mass of organics in vent stream exiting the control device during run j , kg.

T = Total time of all runs, hour.

n = Number of runs.

(4) The organic reduction efficiency for the control device shall be calculated as follows:

$$R = \frac{E_a - E_v}{E_a} \times 100$$

Where:

R = Total organic reduction efficiency for the control device, percent.

E_a = Mass flow rate of organics entering the control device, kg/hr.

E_v = Mass flow rate of organics exiting the control device, kg/hr.

[55 FR 8346, Mar. 7, 1990; 55 FR 12444, Apr. 3, 1990]

§ 61.356 Recordkeeping requirements.

(a) Each owner or operator of a facility subject to the provisions of this subpart shall comply with the recordkeeping requirements of this section. Each record shall be maintained in a readily accessible location at the facility site for a period not less than two years from the date the information is recorded unless otherwise specified.

(b) Each owner or operator shall maintain records that identify each waste stream at the facility subject to this subpart, and indicate whether or not the waste stream is controlled for benzene emissions in accordance with this subpart. In addition the owner or operator shall maintain the following records:

(1) For each waste stream not controlled for benzene emissions in accordance with this subpart, the records shall include all test results, measurements, calculations, and other documentation used to determine the following information for the waste stream: waste stream identification, water content, whether or not the waste stream is a process wastewater stream, annual waste quantity, range of benzene concentrations, annual average flow-weighted benzene concentration, and annual benzene quantity.

(2) For each process wastewater stream not controlled for benzene emissions in accordance with § 61.342(c)(3) of this subpart, the records shall include all measurements, calculations, and other documentation used to determine that the continuous flow of process wastewater is less than 0.02 liters per minute or the annual waste quantity of process wastewater is less than 10 Mg/yr.

(3) For each facility where process wastewater streams are controlled for benzene emissions in accordance with § 61.342(d) of this subpart, the records shall include for each treated process wastewater stream all measurements, calculations, and other documentation used to determine the annual benzene quantity in the process wastewater stream exiting the treatment process.

(4) For each facility where wastewater streams are controlled for

benzene emissions in accordance with § 61.348(b)(1)(i) of this subpart, the records shall include all measurements, calculations, and other documentation used to determine the annual benzene quantity in the wastewater streams exiting wastewater treatment systems at the facility.

(c) An owner or operator transferring waste off site to another facility for treatment in accordance with § 61.342(e) of this subpart shall maintain documentation for each offsite waste shipment that includes the following information: date waste is shipped offsite, quantity of waste shipped offsite, name and address of the facility receiving the waste, and a copy of the notice sent with the waste shipment.

(d) An owner or operator using control equipment in accordance with § 61.343 through 61.347 of this subpart shall maintain engineering design documentation for all control equipment that is installed on the waste management unit. The documentation shall be retained for the life of the control equipment. If a cover is used, then the documentation shall include the following information: cover type, name of company manufacturing or fabricating the cover, manufacturer model number, cover dimensions, materials used to fabricate cover, mechanism used to install cover on the waste management unit and seal the cover perimeter; type, dimensions, and location of each opening; and mechanism used to close and seal each opening. If a control device is used, then the owner or operator shall maintain the control device records required by paragraph (f) of this section.

(e) An owner or operator using a treatment process or wastewater treatment system unit in accordance with § 61.348 of this subpart shall maintain the following records. The documentation shall be retained for the life of the unit.

(1) A statement signed and dated by the owner or operator certifying that the unit is designed to operate at the documented performance level when the waste stream entering the unit is at the highest waste stream flow rate

and benzene content expected to occur.

(2) If engineering calculations are used to determine treatment process or wastewater treatment system unit performance, then the owner or operator shall maintain the complete design analysis for the unit. The design analysis shall include the following information: a list of all information references and sources used in preparing the documentation; design specifications, drawings, schematics, and piping and instrumentation diagrams; and other documentation necessary to demonstrate the unit performance.

(3) If performance tests are used to determine treatment process or wastewater treatment system unit performance, then the owner or operator shall maintain all test information necessary to demonstrate the unit performance.

(i) A description of the unit including the following information: type of treatment process; manufacturer name and model number; and for each waste stream entering and exiting the unit, the waste stream type (e.g., process wastewater, sludge, slurry, etc.), and the design flow rate and benzene content.

(ii) Documentation describing the test protocol and the means by which sampling variability and analytical variability were accounted for in the determination of the unit performance. The description of the test protocol shall include the following information: sampling locations, sampling method, sampling frequency, and analytical procedures used for sample analysis.

(iii) Records of unit operating conditions during each test run including all key process parameters.

(iv) All test results.

(4) If a control device is used, then the owner or operator shall maintain the control device records required by paragraph (f) of this section.

(f) An owner or operator using a closed-vent system and control device in accordance with § 61.349 of this subpart shall maintain the following records. The documentation shall be retained for the life of the control device.

(1) A statement signed and dated by the owner or operator certifying that the closed-vent system and control device is designed to operate at the documented performance level when the waste management unit vented to the control device is or would be operating at the highest load or capacity expected to occur.

(2) If engineering calculations are used to determine control device performance in accordance with § 61.349(c) of this subpart, then a design analysis for the control device that includes:

(i) A list of all information references and sources used in preparing the documentation,

(ii) Specifications, drawings, schematics, and piping and instrumentation diagrams prepared by the owner or operator, or the control device manufacturer or vendor that describe the control device design based on acceptable engineering texts. The design analysis shall address the following vent stream characteristics and control device operating parameters:

(A) For a thermal vapor incinerator, the design analysis shall consider the vent stream composition, constituent concentrations, and flow rate. The design analysis shall also establish the design minimum and average temperature in the combustion zone and the combustion zone residence time.

(B) For a catalytic vapor incinerator, the design analysis shall consider the vent stream composition, constituent concentrations, and flow rate. The design analysis shall also establish the design minimum and average temperatures across the catalyst bed inlet and outlet.

(C) For a boiler or process heater, the design analysis shall consider the vent stream composition, constituent concentrations, and flow rate. The design analysis shall also establish the design minimum and average flame zone temperatures, combustion zone residence time, and description of method and location where the vent stream is introduced into the flame zone.

(D) For a flare, the design analysis shall consider the vent stream composition, constituent concentrations, and flow rate. The design analysis shall

also consider the requirements specified in 40 CFR 60.18.

(E) For a condenser, the design analysis shall consider the vent stream composition, constituent concentrations, flow rate, relative humidity, and temperature. The design analysis shall also establish the design outlet organic compound concentration level, design average temperature of the condenser exhaust vent stream, and the design average temperatures of the coolant fluid at the condenser inlet and outlet.

(F) For a carbon adsorption system that regenerates the carbon bed directly on-site in the control device such as a fixed-bed adsorber, the design analysis shall consider the vent stream composition, constituent concentrations, flow rate, relative humidity, and temperature. The design analysis shall also establish the design exhaust vent stream organic compound concentration level, number and capacity of carbon beds, type and working capacity of activated carbon used for carbon beds, design total steam flow over the period of each complete carbon bed regeneration cycle, duration of the carbon bed steaming and cooling/drying cycles, design carbon bed temperature after regeneration, design carbon bed regeneration time, and design service life of carbon.

(G) For a carbon adsorption system that does not regenerate the carbon bed directly on-site in the control device such as a carbon canister, the design analysis shall consider the vent stream composition, constituent concentrations, flow rate, relative humidity, and temperature. The design analysis shall also establish the design exhaust vent stream organic compound concentration level, capacity of carbon bed, type and working capacity of activated carbon used for carbon bed, and design carbon replacement interval based on the total carbon working capacity of the control device and source operating schedule.

(3) If performance tests are used to determine control device performance in accordance with § 61.349(c) of this subpart:

(i) A description of how it is determined that the test is conducted when the waste management unit or treatment process is operating at the high-

est load or capacity level. This description shall include the estimated or design flow rate and organic content of each vent stream and definition of the acceptable operating ranges of key process and control parameters during the test program.

(ii) A description of the control device including the type of control device, control device manufacturer's name and model number, control device dimensions, capacity, and construction materials.

(iii) A detailed description of sampling and monitoring procedures, including sampling and monitoring locations in the system, the equipment to be used, sampling and monitoring frequency, and planned analytical procedures for sample analysis.

(iv) All test results.

(g) An owner or operator shall maintain a record for each visual inspection required by §§ 61.343 through 61.347 of this subpart that identifies a problem (such as a broken seal, gap or other problem) which could result in benzene emissions. The record shall include the date of the inspection, waste management unit and control equipment location where the problem is identified, a description of the problem, a description of the corrective action taken, and the date the corrective action was completed.

(h) An owner or operator shall maintain a record for each test of no detectable emissions required by §§ 61.343 through 61.347 and § 61.349 of this subpart. The record shall include the following information: date the test is performed, background level measured during test, and maximum concentration indicated by the instrument reading measured for each potential leak interface. If detectable emissions are measured at a leak interface, then the record shall also include the waste management unit, control equipment, and leak interface location where detectable emissions were measured, a description of the problem, a description of the corrective action taken, and the date the corrective action was completed.

(i) For each treatment process and wastewater treatment system unit operated to comply with § 61.348, the owner or operator shall maintain doc-

umentation that includes the following information regarding the unit operation:

(1) Dates of startup and shutdown of the unit.

(2) If measurements of waste stream benzene concentration are performed in accordance with § 61.354(a)(1) of this subpart, the owner or operator shall maintain records that include date each test is performed and all test results.

(3) If a process parameter is continuously monitored in accordance with § 61.354(a)(2) of this subpart, the owner or operator shall maintain records that include a description of the operating parameter (or parameters) to be monitored to ensure that the unit will be operated in conformance with these standards and the unit's design specifications, and an explanation of the criteria used for selection of that parameter (or parameters). This documentation shall be kept for the life of the unit.

(4) Periods when the unit is not operated as designed.

(j) For each control device, the owner or operator shall maintain documentation that includes the following information regarding the control device operation:

(1) Dates of startup and shutdown of the closed-vent system and control device.

(2) A description of the operating parameter (or parameters) to be monitored to ensure that the control device will be operated in conformance with these standards and the control device's design specifications and an explanation of the criteria used for selection of that parameter (or parameters). This documentation shall be kept for the life of the control device.

(3) Periods when the closed-vent system and control device is not operated as designed including periods when a flare pilot does not have a flame.

(4) If a thermal vapor incinerator is used, then the owner or operator shall maintain continuous records of the temperature of the gas stream in the combustion zone of the incinerator and records of all 3-hour periods of operation during which the average temperature of the gas stream in the com-

bustion zone is more than 28 °C below the design combustion zone temperature.

(5) If a catalytic vapor incinerator is used, then the owner or operator shall maintain continuous records of the temperature of the gas stream both upstream and downstream of the catalyst bed of the incinerator, records of all 3-hour periods of operation during which the average temperature measured before the catalyst bed is more than 28 °C below the design gas stream temperature, and records of all 3-hour periods of operation during which the average temperature difference across the catalyst bed is less than 80 percent of the design temperature difference.*

(6) If a boiler or process heater is used, then the owner or operator shall maintain records of each occurrence when there is a change in the location at which the vent stream is introduced into the flame zone as required by § 61.349(a)(2)(i)(C) of this subpart. For a boiler or process heater having a design heat input capacity less than 44 MW, the owner or operator shall maintain continuous records of the temperature of the gas stream in the combustion zone of the boiler or process heater and records of all 3-hour periods of operation during which the average temperature of the gas stream in the combustion zone is more than 28 °C below the design combustion zone temperature. For a boiler or process heater having a design heat input capacity greater than or equal to 44 MW, the owner or operator shall maintain continuous records of the parameter(s) monitored in accordance with the requirements of § 61.354(b)(5) of this subpart.

(7) If a flare is used, then the owner or operator shall maintain continuous records of the flare pilot flame monitoring and records of all periods during which the pilot flame is absent.

(8) If a condenser is used, then the owner or operator shall maintain continuous records of the parameters selected to be monitored in accordance with § 61.354(c)(6) of this subpart. If concentration of organics in the control device outlet gas stream is monitored, then the owner or operator shall record all 3-hour periods of operation during which the concentration

of organics in the exhaust stream is more than 20 percent greater than the design value. If the temperature of the condenser exhaust stream and coolant fluid is monitored, then the owner or operator shall record all 3-hour periods of operation during which the temperature of the condenser exhaust vent stream is more than 6 °C above the design average exhaust vent stream temperature, or the temperature of the coolant fluid existing the condenser is more than 6 °C above the design average coolant fluid temperature at the condenser outlet.

(9) If a carbon adsorber is used, then the owner or operator shall maintain continuous records of the concentration of organics in the control device outlet gas stream. If concentration of organics in the control device outlet gas stream is monitored, then the owner or operator shall record all 3-hour periods of operation during which the concentration of organics in the exhaust stream is more than 20 percent greater than the design value. If the carbon bed regeneration interval is monitored, then the owner or operator shall record each occurrence when the vent stream continues to flow through the control device beyond the predetermined carbon bed regeneration time.

(10) If a carbon adsorber that is not regenerated directly on site in the control device is used, then the owner or operator shall maintain records of dates and times when the control device is monitored, when breakthrough is measured, and shall record the date and time then the existing carbon in the control device is replaced with fresh carbon.

(11) If an alternative operational or process parameter is monitored for a control device, as allowed in § 61.354(b) of this subpart, then the owner or operator shall maintain records of the continuously monitored parameter, including periods when the device is not operated as designed.

(k) An owner or operator who elects to install and operate the control equipment in § 61.351 of this subpart shall comply with the recordkeeping requirements in 40 CFR 60.115b.

(l) An owner or operator who elects to install and operate the control

equipment in § 61.352 of this subpart shall maintain records of the following:

(1) The date, location, and corrective action for each visual inspection required by 40 CFR 60.693-2(a)(5), during which a broken seal, gap, or other problem is identified that could result in benzene emissions.

(2) Results of the seal gap measurements required by 40 CFR 60.693-2(a).

(Approved by the Office of Management and Budget under control number 2060-0183)

[55 FR 8346, Mar. 7, 1990; 55 FR 12444, Apr. 3, 1990; 55 FR 18331, May 2, 1990]

§ 61.357 Reporting requirements.

(a) Each owner or operator subject to this subpart shall submit to the Administrator within 90 days after the effective date of this subpart, or by the initial startup for a new source with an initial startup after the effective date, a report that summarizes the regulatory status of each waste stream subject to this subpart and is determined by the procedures specified in § 61.355(c) of this subpart to contain benzene. The report shall include the following information:

(1) Total annual benzene quantity from facility waste determined in accordance with § 61.355(a) of this subpart.

(2) A table identifying each waste stream and whether or not the waste stream will be controlled for benzene emissions in accordance with the requirements of this subpart.

(3) For each waste stream identified as not being controlled for benzene emissions in accordance with the requirements of this subpart the following information shall be added to the table:

(i) Whether or not the water content of the waste stream is greater than 10 percent;

(ii) Whether or not the waste stream is a process wastewater stream, product tank drawdown, or landfill leachate;

(iii) Annual waste quantity for the waste stream;

(iv) Range of benzene concentrations for the waste stream;

(v) Annual average flow-weighted benzene concentration for the waste stream; and

(vi) Annual benzene quantity for the waste stream.

(b) If the total annual benzene quantity from facility waste is less than 1 Mg/yr, then the owner or operator shall submit to the Administrator a report that updates the information listed in paragraphs (a)(1) through (a)(3) of this section whenever there is a change in the process generating the waste stream that could cause the total annual benzene quantity from facility waste to increase to 10 Mg/yr or more.

(c) If the total annual benzene quantity from facility waste is less than 10 Mg/yr but is equal to or greater than 1 Mg/yr, then the owner or operator shall submit to the Administrator a report that updates the information listed in paragraphs (a)(1) through (a)(3) of this section. The report shall be submitted annually and whenever there is a change in the process generating the waste stream that could cause the total annual benzene quantity from facility waste to increase to 10 Mg/yr or more.

(d) If the total annual benzene quantity from facility waste is equal to or greater than 10 Mg/yr, then the owner or operator shall submit to the Administrator the following reports:

(1) Within 2 years after March 7, 1990, or by the date of initial startup for a new source with an initial startup after the effective date, a certification that the equipment necessary to comply with these standards has been installed and that the required initial inspections or tests have been carried out in accordance with this subpart.

(2) Beginning on the date that the equipment necessary to comply with these standards has been certified in accordance with paragraph (d)(1) of this section, the owner or operator shall submit annually to the Administrator a report that updates the information listed in paragraphs (a)(1) through (a)(3) of this section.

(3) If an owner or operator elects to comply with the alternative requirements of § 61.342(d) of this subpart, then he shall include in the report required by paragraph (d)(2) of this section

tion a table presenting the following information for each process wastewater stream:

(i) Whether or not the process wastewater stream is being controlled for benzene emissions in accordance with the requirements of this subpart;

(ii) For each process wastewater stream identified as not being controlled for benzene emissions in accordance with the requirements of this subpart, the table shall report the following information for the process wastewater stream as determined at the point of waste generation: annual waste quantity, range of benzene concentrations, annual average flow-weighted benzene concentration, and annual benzene quantity;

(iii) For each process wastewater stream identified as being controlled for benzene emissions in accordance with the requirements of this subpart, the table shall report the following information for the process wastewater stream as determined at the exit to the treatment process: Annual waste quantity, range of benzene concentrations, annual average flow-weighted benzene concentration, and annual benzene quantity.

(4) If an owner or operator complies with the requirements of § 61.348(b) of this subpart, then he shall include in the report required by paragraph (d)(2) of this section a table presenting the annual benzene quantity in each wastewater stream exiting wastewater treatment systems at the facility.

(5) Beginning 3 months after the date that the equipment necessary to comply with these standards has been certified in accordance with paragraph (d)(1) of this section, the owner or operator shall submit quarterly to the Administrator a certification that all of the required inspections have been carried out in accordance with the requirements of this subpart.

(6) Beginning 3 months after the date that the equipment necessary to comply with these standards has been certified in accordance with paragraph (d)(1) of this section, the owner or operator shall submit a report quarterly to the Administrator that includes:

(i) If a treatment process or wastewater treatment system unit is monitored in accordance with

§ 61.354(a)(1) of this subpart, then each period of operation during which the concentration of benzene in the monitored waste stream exiting the unit is equal to or greater than 10 ppmw.

(ii) If a treatment process or wastewater treatment system unit is monitored in accordance with § 61.354(a)(2) of this subpart, then each 3-hour period of operation during which the average value of the monitored parameter is outside the range of acceptable values or during which the unit is not operating as designed.

(iii) For a control device monitored in accordance with § 61.354(c) of this subpart, each period of operation monitored during which any of the following conditions occur, as applicable to the control device:

(A) Each 3-hour period of operation during which the average temperature of the gas stream in the combustion zone of a thermal vapor incinerator, as measured by the temperature monitoring device, is more than 28°C below the design combustion zone temperature.

(B) Each 3-hour period of operation during which the average temperature of the gas stream immediately before the catalyst bed of a catalytic vapor incinerator, as measured by the temperature monitoring device, is more than 28°C below the design gas stream temperature, and any 3-hour period during which the average temperature difference across the catalyst bed (i.e., the difference between the temperatures of the gas stream immediately before and after the catalyst bed), as measured by the temperature monitoring device, is less than 80 percent of the design temperature difference.

(C) Each 3-hour period of operation during which the average temperature of the gas stream in the combustion zone of a boiler or process heater having a design heat input capacity less than 44 MW, as measured by the temperature monitoring device, is more than 28°C below the design combustion zone temperature.

(D) Each 3-hour period of operation during which the average concentration of organics in the exhaust gases from a carbon adsorber, condenser, or other vapor recovery system is more

than 20 percent greater than the design exhaust gas concentration level.

(E) Each 3-hour period of operation during which the temperature of the condenser exhaust vent stream is more than 6°C above the design average exhaust vent stream temperature, or the temperature of the coolant fluid exiting the condenser is more than 6°C above the design average coolant fluid temperature at the condenser outlet.

(F) Each period in which the pilot flame of a flare is absent.

(G) Each occurrence when there is a change in the location at which the vent stream is introduced into the flame zone of a boiler or process heater as required by § 61.349(a)(2)(i)(C) of this subpart.

(H) Each occurrence when the carbon in a carbon adsorber system that is regenerated directly on site in the control device is not regenerated at the predetermined carbon bed regeneration time.

(I) Each occurrence when the carbon in a carbon adsorber system that is not regenerated directly on site in the control device is not replaced at the predetermined interval specified in § 61.354(c) of this subpart.

(7) Beginning one year after the date that the equipment necessary to comply with these standards has been certified in accordance with paragraph (d)(1) of this section, the owner or operator shall submit annually to the Administrator a report that summarizes all inspections required by §§ 61.342 through 61.352 of this subpart during which detectable emissions are measured or a problem (such

as a broken seal, gap or other problem) that could result in benzene emissions is identified, including information about the repairs or corrective action taken.

(e) An owner or operator electing to comply with the provisions of §§ 61.351 or 61.352 of this subpart shall notify the Administrator of the alternative standard selected in the report required under § 61.07 or § 61.10 of this part.

(f) An owner or operator who elects to install and operate the control equipment in § 61.351 of this subpart shall comply with the reporting requirements in 40 CFR 60.115b.

(g) An owner or operator who elects to install and operate the control equipment in § 61.352 of this subpart shall submit initial and quarterly reports that identify all seal gap measurements, as required in 40 CFR 60.693-2(a), that are outside the prescribed limits.

(Approved by the Office of Management and Budget under control number 2060-0183)

[55 FR 8346, Mar. 7 1990; 55 FR 12444, Apr. 3, 1990]

§ 61.358 Delegation of authority.

(a) In delegating implementation and enforcement authority to a State under section 112(d) of the Clean Air Act, the authorities contained in paragraph (b) of this section shall be retained by the Administrator and not transferred to a State.

(b) Alternative means of emission limitation under § 61.353 of this subpart will not be delegated to States.

APPENDIX A

National Emission Standards for Hazardous Air Pollutants

Compliance Status Information

1. SOURCE REPORT

INSTRUCTIONS: Owners or operators of sources of hazardous pollutants subject to the National Emission Standards for Hazardous Air Pollutants are required to submit the information contained in Section I to the appropriate U.S. Environmental Protection Agency Regional Office prior to 90 days after the effective date of any standards or amendments which require the submission of such information.

A list of regional offices is provided in §61.04.

A. SOURCE INFORMATION1. Identification/Location - Indicate the name and address of each source.

1 Region	2	3 State	4	5 County	8	9 Source Number	13	0 0 0	0 0	1
								14	16	17 18 19
20 AQCR #	22	23 City Code	26	27 Source Name				46		
				47 Street Address (Location of Plant)				66 80		
Dup 1-18		19	20 City Name				34	State	35	39
				40 State Regis. Number				54	55 NEDS X Ref.	58
				59 SIC				62	8 FF	77 A/P
								64	65	79 Staff
Dup 1-10		5 19	25 30	STP 31	EC 49	80				

2. Contact - Indicate the name and telephone number of the owner or operator or other responsible official whom EPA may contact concerning this report.

Dup 1-18 ^{4 1}
19 20 21 _____ Name _____ 43

^{44 46}
Area Code 47 Number 54 80

3. Source Description - Briefly state the nature of the source (e.g., "Chlor-alkali Plant" or "Machine Shop").

Dup 1-18 ^{4 2}
19 20 21 _____ Description _____ 50

51 _____ Continued _____ 79 80

4. Alternative Mailing Address - Indicate an alternative mailing address if correspondence is to be directed to a location different than that specified above.

Dup 1-18 ^{4 3}
19 20 21 Number Street or Box Number 45 80

Dup 1-18 ^{4 4}
19 20 21 City 35 ^{37 38} State 41 Zip 44 W

5. Compliance Status - The emissions from this source can ☐ cannot meet the emission limitations contained in the National Emission Standards on or prior to 90 days after the effective date of any standards or amendments which require the submission of such information.

Signature of Owner, Operator or Other Responsible Official

NOTE: If the emissions from the source will exceed those limits set by the National Emission Standards for Hazardous Air Pollutants, the source will be in violation and subject to Federal enforcement actions unless granted a waiver of compliance by the Administrator of the U.S. Environmental Protection Agency. The information needed for such waivers is listed in Section II of this form.

- B. PROCESS INFORMATION. Part B should be completed separately for each point of emission for each hazardous pollutant. [Sources subject to 61.22(i) may omit number 4. below.]

Dup 1-13 ^{0 0 5}
14 16 17 18 19 20 SCC 27 28 29 30 31
NEDS X Ref LS SIP

1. **Pollutant Emitted** - Indicate the type of hazardous pollutant emitted by the process. Indicate "AB" for asbestos, "BE" for beryllium, or "HG" for mercury.

32 33
Pollutant

34 Regulation 48

49
EC

2. **Process Description** - Provide a brief description of each process (e.g., "hydrogen end box" in a mercury chlor-alkali plant, "grinding machine" in a beryllium machine shop). Use additional sheets if necessary.

50 Process Description 74

80

Dup 1-18

6 1

19 20

21

50

51

79 80

Dup 1-18

6 2

19 20

21

50

51

79 80

3. **Amount of Pollutant** - Indicate the average weight of the hazardous material named in Item 1 which enters the process in pounds per month (based on the previous twelve months of operation).

Dup 1-18

6 3

19 20

21

27

lbs./mo.

29

36

80

4. **Control Devices**

- a. Indicate the type of pollution control devices, if any, used to reduce the emissions from the process (e.g., venturi scrubber, baghouse, wet cyclone) and the estimated percent of the pollutant which the device removes from the process gas stream.

Dup 1-18

6 4

19 20

21

PRIMARY CONTROL DEVICE:

43

45 Primary Device Name 64

66

70

Percent Removal
Efficiency

72

79

80

Dup 1-18	6 5	SECONDARY CONTROL DEVICES:	45
	19 20	21	
47	Secondary Device Name		64
	Percent Removal Efficiency		70
			72
			79 80

b. Asbestos Emission Control Devices Only

1. If a baghouse is specified in Item 4a, give the following information:

• The air flow permeability in cubic feet per minute per square foot of fabric area.

Air flow permeability = _____ cfm/ft²

• The pressure drop in inches water gauge across the filter at which the baghouse is operated.

Operating pressure drop = _____ inches w.g.

• If the baghouse material contains synthetic fill yarn, check whether this material is / / spun / / or not spun.

• If the baghouse utilizes a felted fabric, give the minimum thickness in inches and the density in ounces per square yard.

Thickness = _____ inches Density = _____ oz/yd²

11. If a wet collection device is specified in Item 4a, give the designed unit contacting energy in inches water gauge.

• Unit contacting energy = _____ inches w.g.

C. DISPOSAL OF ASBESTOS-CONTAINING WASTES. Part C should be completed separately for each asbestos-containing waste generation operation arising from sources subject to §61.22(a), (c), (e), and (h).

Dup 1-13	0 0	5	20	SCC	27	28 29	30 31
	14 16	17 18	19			NEDS X Ref	CS SIP
A B	Regulation			48	49		
32 33				34	EC		
Pollutant							

Environmental Protection Agency

Pt. 61, App. A

1. Waste Generation - Provide a brief description of each process that generates asbestos-containing waste (e.g. disposal of control device wastes).

50 _____ Process Description _____ 79 80

2. Asbestos Concentration - Indicate the average percentage asbestos content of these materials.

Dup 1-18 6 1 ASBESTOS CONCENTRATION: 43 45 48
19 20 21

50 80

3. Amount of Wastes - Indicate the average weight of asbestos-containing wastes disposed of, measured in kg./day.

Dup 1-18 6 2 kg/day 34 80
19 20 21 27 29

4. Control Methods - Indicate the emission control methods used in all stages of waste disposal, from collection, processing, and packaging to transporting and deposition.

Dup 1-18 6 3 Primary Control Method 43
19 20 21

45 _____ 79 80

Dup 1-18 6 4 _____ 50
19 20 21

51 _____ 79 80

5. Waste Disposal - Indicate the type of disposal site (sanitary landfill, open, covered) or incineration site (municipal, private) where the waste is disposed of and who operates the site (company, private, municipal). State the name and location of the site (closest city or town, county, state).

Dup 1-18 6 5 TYPE OF SITE: 33 35 50
19 20 21

51 _____ 79 80

Dup 1-18 6 6 OPERATOR: 21 29 31 50

51 79 80

Dup 1-18 6 7 LOCATION: 21 29

31 70

71 79 80

- D. WASTE DISPOSAL SITES. Part D should be completed separately for each asbestos waste disposal site subject to section 61.22(1).

Dup 1-13 14 16 17 18 19 20 5 SCC 27 28 29 30 31
MEDS X Ref CS SIP

A B
32 33 34 Regulation 48 49
Pollutant EC

50 WASTE DISPOSAL SITE 68 80

1. Description - Provide a brief description of the site, including its size and configuration, and the distance to the closest city or town, closest residence, and closest primary road.

Dup 1-18 6 1 SITE DESCRIPTION 21 37 39 50

51 79 80

Dup 1-18 6 2 DISTANCE: 21 29 30 TOWN: 34 36 40 42 43 K M

45 RESIDENCE: 54 56 60 62 63 K M ROAD: 65 69 71 75

K M
77 78 80

2. Inactivation - After the site is inactivated, indicate the method or methods used to comply with the standard and send a list of the actions that will be undertaken to maintain the inactivated site.

Dup 1-18	6 8	COMPLIANCE		
	19 20	METHOD/INACTIVE SITE:		
	21			52
54		79		80

II. WAIVER REQUESTS

A. Waiver of Compliance. Owners or operators of sources unable to operate in compliance with the National Emission Standards for Hazardous Air Pollutants prior to 90 days after the effective date of any standards or amendments which require the submission of such information may request a waiver of compliance from the Administrator of the U.S. Environmental Protection Agency for the time period necessary to install appropriate control devices or make modifications to achieve compliance. The Administrator may grant a waiver of compliance with the standard for a period not exceeding two years from the effective date of the hazardous pollutant standards, if he finds that such period is necessary for the installation of controls and that steps will be taken during the period of the waiver to assure that the health of persons will be protected from imminent endangerment.

The report information provided in Section I must accompany this application. Applications should be sent to the appropriate EPA regional office.

1. Processes Involved—Indicate the process or processes emitting hazardous pollutants to which emission controls are to be applied.

2. Controls

a. Describe the proposed type of control device to be added or modification to be made to the process to reduce the emission of hazardous pollutants to an acceptable level. (Use additional sheets if necessary.)

b. Describe the measures that will be taken during the waiver period to assure that the health of persons will be protected from imminent endangerment. (Use additional sheets if necessary.)

3. Increments of Progress—Specify the dates by which the following increments of progress will be met.

Date by which contracts for emission control systems or process modifications will be awarded; or date by which orders will be issued for the purchase of the component parts to accomplish emission control or process modification.

Dup 1-16 $\frac{0\ 1\ 7}{17\ 19}$ 53 54 55 _____ 60 61 MO/DY/YR 66 80

- Date of initiation of on-site construction or installation of emission control equipment or process change.

Dup 1-16 $\frac{0\ 2\ 7}{17\ 19}$ 53 54 55 _____ 60 61 MO/DY/YR 66 80

- Date by which on-site construction or installation of emission control equipment or process modification is to be completed.

Dup 1-16 $\frac{0\ 3\ 7}{17\ 19}$ 53 54 55 _____ 60 61 MO/DY/YR 66 80

- Date by which final compliance is to be achieved.

Dup 1-16 $\frac{0\ 4\ 7}{17\ 19}$ 53 54 55 _____ 60 61 MO/DY/YR 66 80

B. Waiver of Emission Tests. A waiver of emission testing may be granted to owners or operators of sources subject to emission testing if, in the judgment of the Administrator of the Environmental Protection Agency the emissions from the source comply with the appropriate standard or if the owners or operators of the source have requested a waiver of compliance or have been granted a waiver of compliance.

This application should accompany the report information provided in Section I.

1. *Reason*—State the reasons for requesting a waiver of emission testing. If the reason stated is that the emissions from the source are within the prescribed limits, documentation of this condition must be attached.

Date _____

Signature of the owner or operator _____

(Sec. 114, of the Clean Air Act as amended (42 U.S.C. 7414))

[40 FR 48303, Oct. 14, 1975, as amended at 43 FR 8800, Mar. 3, 1978; 50 FR 46295, Sept. 9, 1985]

APPENDIX B—TEST METHODS

Method 101—Determination of particulate and gaseous mercury emissions from chlor-alkali plants—air streams

Method 101A—Determination of particulate and gaseous mercury emissions from sewage sludge incinerators

Method 102—Determination of particulate and gaseous mercury emissions from chlor-alkali plants—hydrogen streams

Method 103—Beryllium screening method

Method 104—Determination of beryllium emissions from stationary sources

Method 105—Determination of mercury in wastewater treatment plant sewage sludges

Method 106—Determination of vinyl chloride from stationary sources

Method 107—Determination of vinyl chloride content of inprocess wastewater samples, and vinyl chloride content of polyvinyl chloride resin, slurry, wet cake, and latex samples

Method 107A—Determination of vinyl chloride content of solvents, resin-solvent solution, polyvinyl chloride resin, resin slurry, wet resin, and latex samples

Method 108—Determination of particulate and gaseous arsenic emissions

Method 108A—Determination of arsenic content in ore samples from nonferrous smelters

Method 108B—Determination of arsenic content in ore samples from nonferrous smelters

Method 108C—Determination of arsenic content in ore samples from nonferrous smelters

Method 111—Determination of Polonium-210 emissions from stationary sources

METHOD 101—DETERMINATION OF PARTICULATE AND GASEOUS MERCURY EMISSIONS FROM CHLOR-ALKALI PLANTS—AIR STREAMS

1. *Applicability and Principle*

1.1 *Applicability.* This method applies to the determination of particulate and gaseous

ous mercury (Hg) emissions from chlor-alkali plants and other sources (as specified in the regulations), where the carrier-gas stream in the duct or stack is principally air.

1.2 Principle. Particulate and gaseous Hg emissions are withdrawn isokinetically from the source and collected in acidic iodine monochloride (ICl) solution. The Hg collected (in the mercuric form) is reduced to elemental Hg, which is then aerated from the solution into an optical cell and measured by atomic absorption spectrophotometry.

2. Range and Sensitivity

2.1 Range. After initial dilution, the range of this method is 0.5 to 120 $\mu\text{g Hg/ml}$. The upper limit can be extended by further dilution of the sample.

2.2 Sensitivity. The sensitivity of this method depends on the recorder/spectrophotometer combination selected.

3. Interfering Agents

3.1 Sampling. SO_2 reduces ICl and causes premature depletion of the ICl solution.

3.2 Analysis. ICl concentrations greater than 10^{-4} molar inhibit the reduction of the Hg (II) ion in the aeration cell. Condensation of water vapor on the optical cell windows causes a positive interference.

4. Precision and Accuracy

The following estimates are based on collaborative tests, wherein 13 laboratories performed duplicate analyses on two Hg-containing samples from a chlor-alkali plant and on one laboratory-prepared sample of known Hg concentration. The concentration ranged from 2 to 65 $\mu\text{g Hg/ml}$.

4.1 Precision. The estimated within-laboratory and between-laboratory standard deviations are 1.6 and 1.8 $\mu\text{g Hg/ml}$, respectively.

4.2 Accuracy. The participating laboratories that analyzed a 64.3- $\mu\text{g Hg/ml}$ (in 0.1 M ICl) standard obtained a mean of 63.7 $\mu\text{g Hg/ml}$.

5. Apparatus

5.1 Sampling Train. A schematic of the sampling train is shown in Figure 101-1; it is similar to the Method 5 train (mention of Method 5 refers to part 60 of 40 CFR). The sampling train consists of the following components:

5.1.1 Probe Nozzle, Pitot Tube, Differential Pressure Gauge, Metering System, Barometer, and Gas Density Determination Equipment. Same as Method 5, Sections 2.1.1, 2.1.3, 2.1.4, 2.1.8, 2.1.9, and 2.1.10, respectively.

5.1.2 Probe Liner. Borosilicate or quartz glass tubing. The tester may use a heating system capable of maintaining a gas temperature of $120 \pm 14^\circ\text{C}$ ($248 \pm 25^\circ\text{F}$) at the probe exit during sampling to prevent water condensation.

NOTE: Do not use metal probe liners.

5.1.3 Impingers. Four Greenburg-Smith impingers connected in series with leak-free ground glass fittings or any similar leak-free noncontaminating fittings. For the first, third, and fourth impingers, the tester may use impingers that are modified by replacing the tip with a 13-mm-ID (0.5-in.) glass tube extending to 13 mm (0.5 in.) from the bottom of the flask.

5.1.4 Acid Trap. Mine Safety Appliances air line filter, Catalog number 81857, with acid absorbing cartridge and suitable connections, or equivalent.

5.2 Sample Recovery. The following items are needed:

5.2.1 Glass Sample Bottles. Leakless, with Teflon-lined caps, 1000- and 100-ml.

5.2.2 Graduated Cylinder. 250-ml.

5.2.3 Funnel and Rubber Policeman. To aid in transfer of silica gel to container; not necessary if silica gel is weighed in the field.

5.2.4 Funnel. Glass, to aid in sample recovery.

5.3 Sample Preparation and Analysis. The following equipment is needed:

5.3.1 Atomic Absorption Spectrophotometer. Perkin-Elmer 303, or equivalent, containing a hollow-cathode mercury lamp and the optical cell described in Section 5.3.2.

5.3.2 Optical Cell. Cylindrical shape with quartz end windows and having the dimensions shown in Figure 101-2. Wind the cell with approximately 2 meters of 24-gauge nichrome heating wire, and wrap with fiberglass insulation tape or equivalent; do not let the wires touch each other.

5.3.3 Aeration Cell. Constructed according to the specifications in Figure 101-3. Do not use a glass frit as a substitute for the blown glass bubbler tip shown in Figure 101-3.

5.3.4 Recorder. Matched to output of the spectrophotometer described in Section 5.3.1.

5.3.5 Variable Transformer. To vary the voltage on the optical cell from 0 to 40 volts.

5.3.6 Hood. For venting optical cell exhaust.

5.3.7 Flowmetering Valve.

5.3.8 Flowmeter. Rotameter or equivalent, capable of measuring a gas flow of 1.5 liters/min.

5.3.9 Aeration Gas Cylinder. Nitrogen or dry, Hg-free air, equipped with a single-stage regulator.

5.3.10 Connecting Tubing. Use glass tubing (ungreased ball- and socket-connections are recommended) for all tubing connections between the solution cell and the optical cell; do not use Tygon tubing, other types of flexible tubing, or metal tubing as substitutes. The tester may use Teflon, steel, or copper tubing between the nitrogen tank and flowmetering valve (5.3.7), and Tygon, gum, or rubber tubing between the flowmetering valve and the aeration cell.

5.3.11 Flow Rate Calibration Equipment. Bubble flowmeter or wet test meter for measuring a gas flow rate of 1.5 ± 0.1 liters/min.

5.3.12 Volumetric Flasks. Class A with penny head standard taper stoppers; 100-, 250-, 500- and 1000-ml.

5.3.13 Volumetric Pipets. Class A; 1-, 2-, 3-, 4-, and 5-ml.

5.3.14 Graduated Cylinder. 50-ml.

5.3.15 Magnetic Stirrer. General-purpose laboratory type.

5.3.16 Magnetic Stirring Bar. Teflon-coated.

5.3.17 Balance. Capable of weighing to ± 0.5 g.

5.4 Alternative Analytical Apparatus. Alternative systems are allowable as long as they meet the following criteria:

5.4.1 A linear calibration curve is generated and two consecutive samples of the same aliquot size and concentration agree within 3 percent of their average.

5.4.2 A minimum of 95 percent of the spike is recovered when an aliquot of a source sample is spiked with a known concentration of mercury (II) compound.

5.4.3 The reducing agent should be added after the aeration cell is closed.

5.4.4 The aeration bottle bubbler should not contain a frit.

5.4.5 Any Tygon used should be as short as possible and conditioned prior to use until blanks and standards yield linear and reproducible results.

5.4.6 If manual stirring is done before aeration, it should be done with the aeration cell closed.

5.4.7 A drying tube should not be used unless it is conditioned as the Tygon above.

6. Reagents

Use ACS reagent-grade chemicals or equivalent, unless otherwise specified.

6.1 Sampling and Recovery. The reagents used in sampling and recovery are as follows:

6.1.1 Water. Deionized distilled, meeting ASTM Specifications for Type I Reagent Water—ASTM Test Method D1193-77 (incorporated by reference—see § 61.18). If high concentrations of organic matter are not expected to be present, the analyst may eliminate the KMnO_4 test for oxidizable organic matter. Use this water in all dilutions and solution preparations.

6.1.2 Nitric Acid (HNO_3), 50 Percent (V/V). Mix equal volumes of concentrated HNO_3 and deionized distilled water, being careful to slowly add the acid to the water.

6.1.3 Silica Gel. Indicating type, 6- to 16-mesh. If previously used, dry at 175°C (350°F) for 2 hours. The tester may use new silica gel as received.

6.1.4 Potassium Iodide (KI) Solution, 25 Percent. Dissolve 250 g of KI in deionized distilled water and dilute to 1 liter.

6.1.5 Iodine Monochloride (ICl) Stock Solution, 1.0 M. To 800 ml of 25 percent KI solution, add 800 ml of concentrated hydrochloric acid (HCl). Cool to room temperature. With vigorous stirring, slowly add 135 g of potassium iodate (KIO_3) and stir until all free iodine has dissolved. A clear orange-red solution occurs when all the KIO_3 has been added. Cool to room temperature and dilute to 1800 ml with deionized distilled water. Keep the solution in amber glass bottles to prevent degradation.

6.1.6 Absorbing Solution, 0.1 M ICl . Dilute 100 ml of the 1.0 M ICl stock solution to 1 liter with deionized distilled water. Keep the solution in amber glass bottles and in darkness to prevent degradation. This reagent is stable for at least 2 months.

6.2 Sample Preparation and Analysis. The reagents needed are listed below:

6.2.1 Tin (II) Solution. Prepare fresh daily and keep sealed when not being used. Completely dissolve 20 g of tin (II) chloride [or 25 g of tin (II) sulfate] crystals (Baker Analyzed reagent grade or any other brand that will give a clear solution) in 25 ml of concentrated HCl . Dilute to 250 ml with deionized distilled water. Do not substitute HNO_3 , H_2SO_4 , or other strong acids for the HCl .

6.2.2 Mercury Stock Solution, 1 mg Hg/ml. Prepare and store all mercury standard solutions in borosilicate glass containers. Completely dissolve 0.1354 g of mercury (II) chloride in 75 ml of deionized distilled water in a 100 ml glass volumetric flask. Add 10 ml of concentrated HNO_3 , and adjust the volume to exactly 100 ml with deionized distilled water. Mix thoroughly. This solution is stable for at least 1 month.

6.2.3 Sulfuric Acid, 5 Percent (V/V). Dilute 25 ml of concentrated H_2SO_4 to 500 ml with deionized distilled water.

6.2.4 Intermediate Mercury Standard Solution, 10 μg Hg/ml. Prepare fresh weekly. Pipet 5.0 ml of the mercury stock solution (6.2.2) into a 500-ml glass volumetric flask and add 20 ml of the 5 percent H_2SO_4 solution. Dilute to exactly 500 ml with deionized distilled water. Thoroughly mix the solution.

6.2.5 Working Mercury Standard Solution, 200 ng Hg/ml. Prepare fresh daily. Pipet 5.0 ml from the "Intermediate Mercury Standard Solution" (6.2.4) into a 250-ml volumetric glass flask. Add 10 ml of the 5 percent H_2SO_4 and 2 ml of the 0.1 M ICl absorbing solution taken as a blank (7.2.3) and dilute to 250 ml with deionized distilled water. Mix thoroughly.

7. Procedure

7.1 Sampling. Because of the complexity of this method, testers should be trained and experienced with the test procedures to assure reliable results. Since the amount of

Hg that is collected generally is small, the method must be carefully applied to prevent contamination or loss of sample.

7.1.1 Pretest Preparation. Follow the general procedure given in Method 5, Section 4.1.1, except omit the directions on the filter.

7.1.2 Preliminary Determinations. Follow the general procedure given in Method 5, Section 4.1.2, except as follows: Select a nozzle size based on the range of velocity heads to assure that it is not necessary to change the nozzle size in order to maintain isokinetic sampling rates below 28 liters/min (1.0 cfm).

Obtain samples over a period or periods that accurately determine the maximum emissions that occur in a 24-hour period. In the case of cyclic operations, run sufficient tests for the accurate determination of the emissions that occur over the duration of the cycle. A minimum sample time of 2 hours is recommended. In some instances, high Hg or high SO₂ concentrations make it impossible to sample for the desired minimum time. This is indicated by reddening (liberation of free iodine) in the first impinger. In these cases, the tester may divide the sample run into two or more subruns to insure that the absorbing solution is not depleted.

7.1.3 Preparation of Sampling Train. Clean all glassware (probe, impingers, and connectors) by rinsing with 50 percent HNO₃, tap water, 0.1 M ICl, tap water, and finally deionized distilled water. Place 100 ml of 0.1 M ICl in each of the first three impingers. Take care to prevent the absorbing solution from contacting any greased surfaces. Place approximately 200 g of preweighed silica gel in the fourth impinger. The tester may use more silica gel, but should be careful to ensure that it is not entrained and carried out from the impinger during sampling. Place the silica gel container in a clean place for later use in the sample recovery. Alternatively, determine and record the weight of the silica gel plus impinger to the nearest 0.5 g.

Install the selected nozzle using a Viton A O-ring when stack temperatures are less than 260° C (500° F). Use a fiberglass string gasket if temperatures are higher. See APTD-0576 (Citation 9 in Section 10) for details. Other connecting systems using either 316 stainless steel or Teflon ferrules may be used. Mark the probe with heat-resistant tape or by some other method to denote the proper distance into the stack or duct for each sampling point. Assemble the train as shown in Figure 101-1, using (if necessary) a very light coat of silicone grease on all ground glass joints. Grease only the outer portion (see APTD-0576) to avoid possibility of contamination by the silicone grease.

NOTE: An empty impinger may be inserted between the third impinger and the silica

gel to remove excess moisture from the sample stream.

After the sampling train has been assembled, turn on and set the probe, if applicable, at the desired operating temperature. Allow time for the temperatures to stabilize. Place crushed ice around the impingers.

7.1.4 Leak-Check Procedures. Follow the leak-check procedures outlined in Method 5, Sections 4.1.4.1 (Pretest Leak Check), 4.1.4.2 (Leak Checks During Sample Run), and 4.1.4.3 (Post-Test Leak Check).

7.1.5 Mercury Train Operation. Follow the general procedure given in Method 5, Section 4.1.5. For each run, record the data required on a data sheet such as the one shown in Figure 101-4.

7.1.6 Calculation of Percent Isokinetic. Same as Method 5, Section 4.1.6.

7.2 Sample Recovery. Begin proper cleanup procedure as soon as the probe is removed from the stack at the end of the sampling period.

Allow the probe to cool. When it can be safely handled, wipe off any external particulate matter near the tip of the probe nozzle and place a cap over it. Do not cap off the probe tip tightly while the sampling train is cooling. Capping would create a vacuum and draw liquid out from the impingers.

Before moving the sampling train to the cleanup site, remove the probe from the train, wipe off the silicone grease, and cap the open outlet of the probe. Be careful not to lose any condensate that might be present. Wipe off the silicone grease from the impinger. Use either ground-glass stoppers, plastic caps, or serum caps to close these openings.

Transfer the probe and impinger assembly to a cleanup area that is clean, protected from the wind, and free of Hg contamination. The ambient air in laboratories located in the immediate vicinity of Hg-using facilities is not normally free of Hg contamination.

Inspect the train before and during assembly, and note any abnormal conditions. Treat the sample as follows:

7.2.1 Container No. 1 (Impinger and Probe). Using a graduated cylinder, measure the liquid in the first three impingers to within ± 1 ml. Record the volume of liquid present (e.g., see Figure 5-3 of Method 5). This information is needed to calculate the moisture content of the effluent gas. (Use only glass storage bottles and graduated cylinders that have been precleaned as in Section 7.1.3.) Place the contents of the first three impingers into a 1000-ml glass sample bottle.

Taking care that dust on the outside of the probe or other exterior surfaces does not get into the sample, quantitatively recover the Hg (and any condensate) from the

probe nozzle, probe fitting, and probe liner as follows: Rinse these components with two 50-ml portions of 0.1 M IC1. Next, rinse the probe nozzle, fitting and liner, and each piece of connecting glassware between the probe liner and the back half of the third impinger with a maximum of 400 ml of deionized distilled water. Add all washings to the 1000-ml glass sample bottle containing the liquid from the first three impingers.

After all washings have been collected in the sample container, tighten the lid on the container to prevent leakage during shipment to the laboratory. Mark the height of the liquid to determine later whether leakage occurred during transport. Label the container to clearly identify its contents.

7.2.2 Container No. 2 (Silica Gel). Note the color of the indicating silica gel to determine whether it has been completely spent and make a notation of its condition. Transfer the silica gel from its impinger to its original container and seal. The tester may use as aids a funnel to pour the silica gel and a rubber policeman to remove the silica gel from the impinger. The small amount of particles that may adhere to the impinger wall need not be removed. Since the gain in weight is to be used for moisture calculations, do not use any water or other liquids to transfer the silica gel. If a balance is available in the field, weigh the spent silica gel (or silica gel plus impinger) to the nearest 0.5 g; record this weight.

7.2.3 Container No. 3 (Absorbing Solution Blank). For a blank, place 50 ml of the 0.1 M IC1 absorbing solution in a 100-ml sample bottle. Seal the container. Use this blank to prepare the working mercury standard solution (6.2.5).

7.3 Sample Preparation. Check the liquid level in each container to see whether liquid was lost during transport. If a noticeable amount of leakage occurred, either void the sample or use methods subject to the approval of the Administrator to account for the losses. Then follow the procedures below:

7.3.1 Container No. 1 (Impinger and Probe). Carefully transfer the contents of Container No. 1 into a 1000-ml volumetric flask and adjust the volume to exactly 1000 ml with deionized distilled water.

7.3.2 Dilutions. Pipet a 2-ml aliquot from the diluted sample from 7.3.1 into a 250-ml volumetric flask. Add 10 ml of 5 percent H_2SO_4 and adjust the volume to exactly 250 ml with deionized distilled water. These solutions are stable for at least 72 hours.

NOTE: The dilution factor will be $250/2$ for this solution.

7.4 Analysis. Calibrate the spectrophotometer and recorder and prepare the calibration curve as described in Sections 8.1 to 8.2.

7.4.1 Mercury Samples. Repeat the procedure used to establish the calibration curve with appropriately sized aliquots (1 to 5 ml) of each of the diluted samples (from Section 7.3.2) until two consecutive peak heights agree within ± 3 percent of their average value. The peak maximum of an aliquot (except the 5-ml aliquot) must be greater than 10 percent of the recorder full scale. If the peak maximum of a 1.0-ml aliquot is off scale on the recorder, further dilute the original source sample to bring the Hg concentration into the calibration range of the spectrophotometer.

Run a blank and standard at least after every five samples to check the spectrophotometer calibration; recalibrate as necessary.

It is also recommended that at least one sample from each stack test be checked by the Method of Standard Additions to confirm that matrix effects have not interfered in the analysis.

7.4.2 Container No. 2 (Silica Gel). Weigh the spent silica gel (or silica gel plus impinger) to the nearest 0.5 g using a balance. (This step may be conducted in the field.)

8. Calibration and Standards

Before use, clean all glassware, both new and used, as follows: brush with soap and water, liberally rinse with tap water, soak for 1 hour in 50 percent HNO_3 , and then rinse with deionized distilled water.

8.1 Flow Calibration. Assemble the aeration system as shown in Figure 101-5. Set the outlet pressure on the aeration gas cylinder regulator to a minimum pressure of 500 mm Hg (10 psi), and use the flowmetering valve and a bubble flowmeter or wet test meter to obtain a flow rate of 1.5 ± 0.1 liters/min through the aeration cell. After the flow calibration is complete, remove the bubble flowmeter from the system.

8.2 Optical Cell Heating System Calibration. Using a 50-ml graduated cylinder, add 50 ml of deionized distilled water to the bottle section of the aeration cell and attach the bottle section to the bubbler section of the cell. Attach the aeration cell to the optical cell; and while aerating at 1.5 liters/min, determine the minimum variable transformer setting necessary to prevent condensation of moisture in the optical cell and in the connecting tubing. (This setting should not exceed 20 volts.)

8.3 Spectrophotometer and Recorder Calibration. The mercury response may be measured by either peak height or peak area.

NOTE: The temperature of the solution affects the rate at which elemental Hg is released from a solution and, consequently, it affects the shape of the absorption curve (area) and the point of maximum absorbance (peak height). Therefore, to obtain

reproducible results, bring all solutions to room temperature before use.

Set the spectrophotometer wavelength at 253.7 nm, and make certain the optical cell is at the minimum temperature that will prevent water condensation. Then set the recorder scale as follows: Using a 50-ml graduated cylinder, add 50 ml of deionized distilled water to the aeration cell bottle and pipet 5.0 ml of the working mercury standard solution into the aeration cell.

NOTE: Always add the Hg-containing solution to the aeration cell after the 50 ml of deionized distilled water.

Place a Teflon-coated stirring bar in the bottle. Before attaching the bottle section to the bubbler section of the aeration cell, make certain that (1) the aeration cell exit arm stopcock (Figure 101-3) is closed (so that Hg will not prematurely enter the optical cell when the reducing agent is being added) and (2) there is no flow through the bubbler. If conditions (1) and (2) are met, attach the bottle section to the bubbler section of the aeration cell. Pipet 5 ml of stannous chloride solution into the aeration cell through the side arm, and immediately stopper the side arm. Stir the solution for 15 sec, turn on the recorder, open the aeration cell exit arm stopcock, and then immediately initiate aeration with continued stirring. Determine the maximum absorbance of the standard and set this value to read 90 percent of the recorder full scale.

8.4 Calibration Curve. After setting the recorder scale, repeat the procedure in Section 8.3 using 0.0-, 1.0-, 2.0-, 3.0-, 4.0-, and 5.0-ml aliquots of the working standard solution (final amount of Hg in the aeration cell is 0, 200, 400, 600, 800, and 1000 ng, respectively). Repeat this procedure on each aliquot size until two consecutive peaks agree within 3 percent of their average value. (Note: To prevent Hg carryover from one sample to another, do not close the aeration gas tank valve and do not disconnect the aeration cell from the optical cell until the recorder pen has returned to the baseline.) It should not be necessary to disconnect the aeration gas inlet line from the aeration cell when changing samples. After separating the bottle and bubbler sections of the aeration cell, place the bubbler section into a 600-ml beaker containing approximately 400 ml of deionized distilled water. Rinse the bottle section of the aeration cell with a stream of deionized distilled water to remove all traces of the tin (II) reducing agent. Also, to prevent the loss of Hg before aeration, remove all traces of the reducing agent between samples by washing with deionized distilled water. It will be necessary, however, to wash the aeration cell parts with concentrated HCl if any of the following conditions occur: (1) A white film appears on any inside surface of the aer-

ation cell, (2) the calibration curve changes suddenly, or (3) the replicate samples do not yield reproducible results.

Subtract the average peak height (or peak area) of the blank (0.0-ml aliquot)—which should be less than 2 percent of recorder full scale—from the averaged peak heights of the 1.0-, 2.0-, 3.0-, 4.0-, and 5.0-ml aliquot standards. If the blank absorbance is greater than 2 percent of full-scale, the probable cause is Hg contamination of a reagent or carry-over of Hg from a previous sample. Plot the corrected peak height of each standard solution versus the corresponding final total Hg weight in the aeration cell (in ng) and draw the best-fit straight line. This line should either pass through the origin or pass through a point no further from the origin than ± 2 percent of the recorder full scale. If the line does not pass through or very near to the origin, check for nonlinearity of the curve and for incorrectly prepared standards.

8.5 Sampling Train Calibration. Calibrate the sampling train components according to the procedures outlined in the following sections of Method 5: Section 5.1 (Probe Nozzle), Section 5.2 (Pitot Tube), Section 5.3 (Metering System), Section 5.4 (Probe Heater), Section 5.5 (Temperature Gauges), Section 5.7 (Barometer). Note that the leak-check described in Section 5.6 of Method 5 applies to this method.

9. Calculations

9.1 Dry Gas Volume. Using the data from this test, calculate $V_{m(std)}$, the dry gas sample volume at standard conditions (corrected for leakage, if necessary) as outlined in Section 6.3 of Method 5.

9.2 Volume of Water Vapor and Moisture Content. Using the data obtained from this test, calculate the volume of water vapor $V_{w(std)}$ and the moisture content B_w of the stack gas. Use Equations 5-2 and 5-3 of Method 5.

9.3 Stack Gas Velocity. Using the data from this test and Equation 2-9 of Method 2, calculate the average stack gas velocity v_s .

9.4 Total Mercury. For each source sample, correct the average maximum absorbance of the two consecutive samples whose peak heights agree within ± 3 percent of their average for the contribution of the solution blank (see Section 8.4). Use the calibration curve and these corrected averages, to determine the final total weight of mercury in nanograms in the aeration cell for each source sample. Correct for any dilutions made to bring the sample in the working range of the spectrophotometer. Then calculate the Hg in μg (m_{Hg}) in the original solution as follows:

$$m_{Hg} = \frac{C_{Hg(ac)}(D.F.)V_s 10^{-3}}{S}$$

Eq. 101-1

where:

$C_{Hg(ac)}$ = Total nanograms of mercury in aliquot analyzed (reagent blank subtracted).

D.F. = Dilution factor for the Hg-containing solution (before adding to the aeration cell; e.g., D.F. = 250/2 if the source samples were diluted as described in Section 7.3.2).

V_s = Solution volume of original sample, 1000 ml for samples diluted as described in Section 7.2.1.

10^{-3} = Conversion factor, $\mu\text{g}/\text{ng}$.

S = Aliquot volume added to aeration cell, ml.

9.5 Mercury Emission Rate. Calculate the Hg emission rate R in g/day for continuous operations using Equation 101-2. For cyclic operations, use only the time per day each stack is in operation. The total Hg emission rate from a source will be the summation of results from all stacks.

$$R = K \frac{m_{Hg} v_g A_s (86,400 \times 10^{-6})}{[V_{m(sta)} + V_{w(sta)}](T_s/P_s)}$$

Eq. 101-2

where:

A_s = Stack cross-sectional area, m^2 (ft^2).

86,400 = Conversion factor, sec/day.

10^{-6} = Conversion factor, g/ μg .

T_s = Absolute average stack gas temperature, $^{\circ}\text{K}$ ($^{\circ}\text{R}$).

P_s = Absolute stack gas pressure, mm Hg (in. Hg).

K = 0.3858 $^{\circ}\text{K}/\text{mm Hg}$ for metric units.

= 17.64 $^{\circ}\text{R}/\text{in. Hg}$ for English units.

v_g = Average gas velocity, m/sec (ft/sec).

$V_{m(sta)}$ = Dry gas sample volume at standard conditions, scm (scf).

$V_{w(sta)}$ = Volume of water vapor at standard conditions, scm (scf).

9.6 Isokinetic Variation and Acceptable Results. Same as Method 5, Sections 6.11 and 6.12, respectively.

9.7 Determination of Compliance. Each performance test consists of three repetitions of the applicable test method. For the purpose of determining compliance with an applicable national emission standard, use the average of the results of all repetitions.

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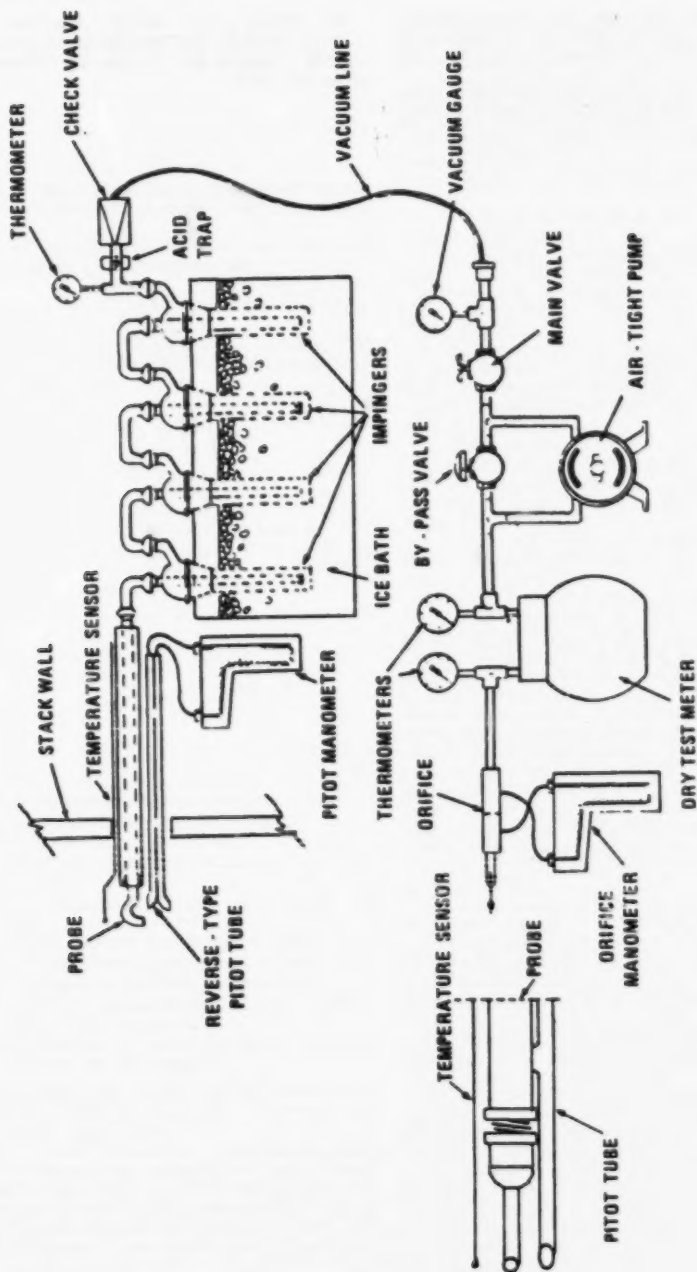


Figure 101-1 Mercury sampling train.

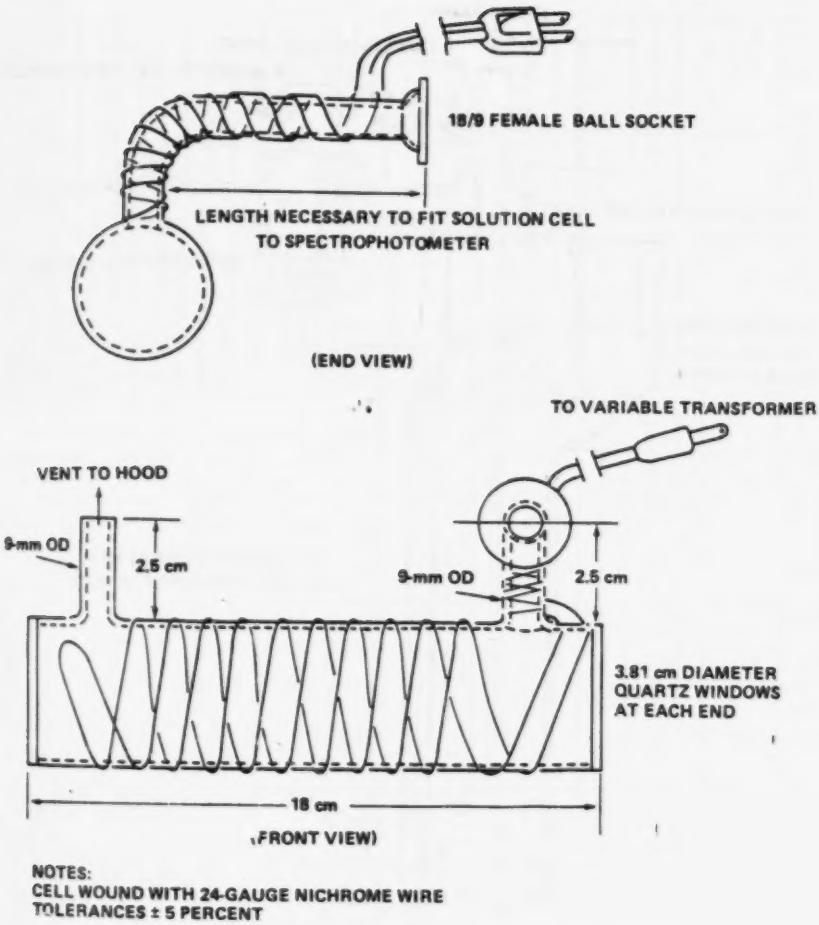


Figure 101-2. Optical cell.

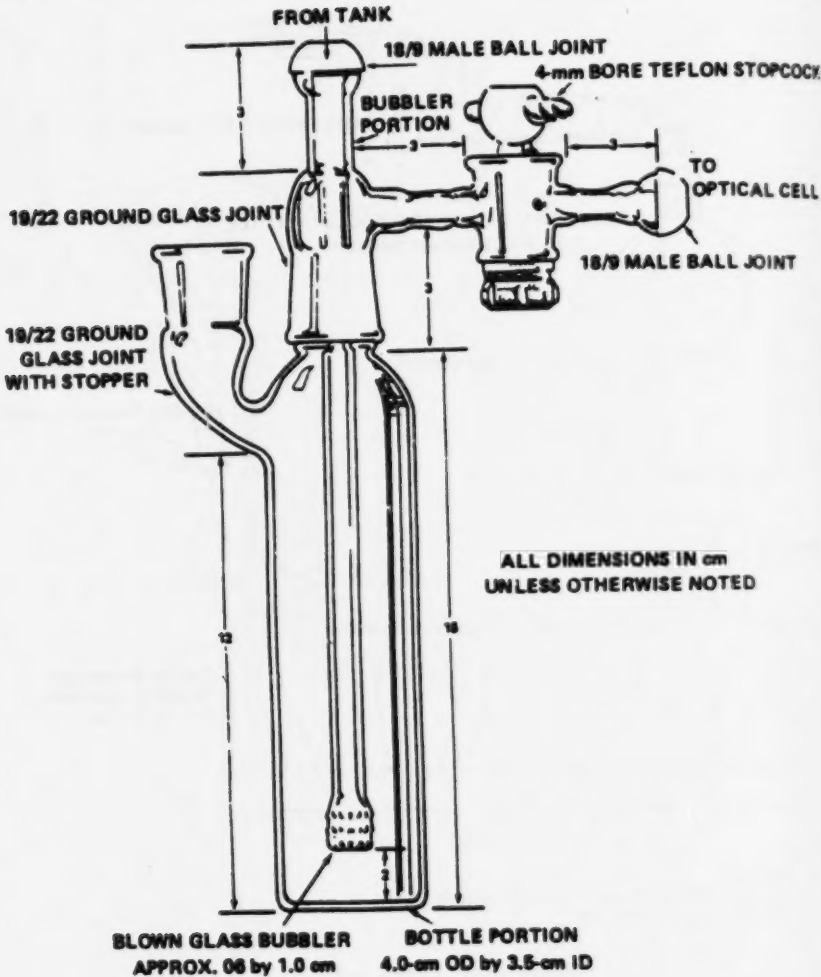


Figure 101-3. Aeration cell.

PLANT _____
LOCATION _____
OPERATOR _____
DATE _____
RUN NO. _____
SAMPLE BOX NO. _____
FILTER BOX NO. _____
METER AMP _____
C FACTOR _____
PITOT TUBE COEFFICIENT, C_p _____

[illegible]

Fig. 101-4. Mercury field data.

IF APPLICABLE

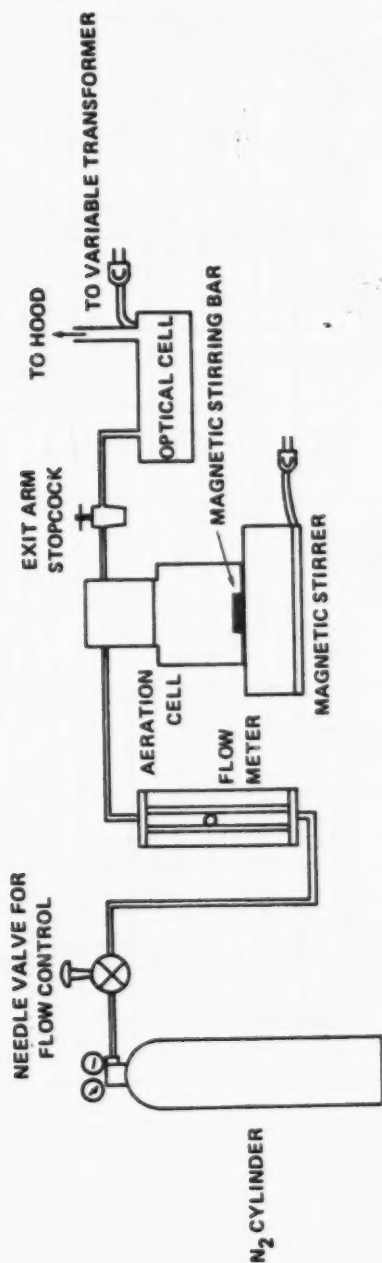


Figure 101-5. Schematic of aeration system.

METHOD 101A—DETERMINATION OF PARTICULATE AND GASEOUS MERCURY EMISSIONS FROM SEWAGE SLUDGE INCINERATORS
METH. 101A

Introduction

This method is similar to Method 101, except acidic potassium permanganate solution is used instead of acidic iodine monochloride for collection.

1. Applicability and Principle

1.1 **Applicability.** This method applies to the determination of particulate and gaseous mercury (Hg) emissions from sewage sludge incinerators and other sources as specified in the regulations.

1.2 **Principle.** Particulate and gaseous Hg emissions are withdrawn isokinetically from the source and collected in acidic potassium permanganate (KMnO₄) solution. The Hg collected (in the mercuric form) is reduced to elemental Hg, which is then aerated from the solution into an optical cell and measured by atomic absorption spectrophotometry.

2. Range and Sensitivity

2.1 **Range.** After initial dilution, the range of this method is 20 to 800 ng Hg/ml. The upper limit can be extended by further dilution of the sample.

2.2 **Sensitivity.** The sensitivity of the method depends on the recorder/spectrophotometer combination selected.

3. Interfering Agents

3.1 **Sampling.** Excessive oxidizable organic matter in the stack gas prematurely depletes the KMnO₄ solution and thereby prevents further collection of Hg.

3.2 **Analysis.** Condensation of water vapor on the optical cell windows causes a positive interference.

4. Precision

Based on eight paired-train tests, the within-laboratory standard deviation was estimated to be 4.8 µg Hg/ml in the concentration range of 50 to 130 µg Hg/m³.

5. Apparatus

5.1 **Sampling Train and Sample Recovery.** Same as Method 101, Sections 5.1 and 5.2, respectively, except for the following variations:

5.1.1 **Probe Liner.** Same as Method 101, Section 5.1.2, except that if a filter is used ahead of the impingers, the tester must use the probe heating system to minimize the condensation of gaseous Hg.

5.1.2 **Filter Holder (Optional).** Borosilicate glass with a rigid stainless-steel wire-screen filter support (do not use glass frit supports) and a silicone rubber or Teflon gasket, designed to provide a positive seal against leakage from outside or around the filter. The filter holder must be equipped with a filter heating system capable of

maintaining a temperature around the filter holder of 120 ± 14° C (248 ± 25° F) during sampling to minimize both water and gaseous Hg condensation. The tester may use a filter in cases where the stream contains large quantities of particulate matter.

5.2 **Analysis.** The apparatus needed for analysis is the same as Method 101, Sections 5.3 and 5.4, except as follows:

5.2.1 **Volumetric Pipets.** Class A; 1-, 2-, 3-, 4-, 5-, 10-, and 20-ml.

5.2.2 **Graduated Cylinder.** 25-ml.

5.2.3 **Steam Bath.**

6. Reagents

Use ACS reagent-grade chemicals or equivalent, unless otherwise specified.

6.1 **Sampling and Recovery.** The reagents used in sampling and recovery are as follows:

6.1.1 **Water.** Deionized distilled, meeting ASTM Specifications for Type I Reagent Water—ASTM Test Method D1193-77 (incorporated by reference—see § 61.18). If high concentrations of organic matter are not expected to be present, the analyst may eliminate the KMnO₄ test for oxidizable organic matter. Use this water in all dilutions and solution preparations.

6.1.2 **Nitric Acid (HNO₃).** 50 Percent (V/V). Mix equal volumes of concentrated HNO₃ and deionized distilled water, being careful to slowly add the acid to the water.

6.1.3 **Silica Gel.** Indicating type, 6- to 16-mesh. If previously used, dry at 175° C (350° F) for 2 hr. The tester may use new silica gel as received.

6.1.4 **Filter (Optional).** Glass fiber filter, without organic binder, exhibiting at least 99.95 percent efficiency on 0.3 µm dioctyl phthalate smoke particles. The tester may use the filter in cases where the gas stream contains large quantities of particulate matter, but he should analyze blank filters for Hg content.

6.1.5 **Sulfuric Acid (H₂SO₄).** 10 Percent (V/V). Add and mix 100 ml of concentrated H₂SO₄ with 900 ml of deionized distilled water.

6.1.6 **Absorbing Solution.** 4 Percent KMnO₄ (W/V). Prepare fresh daily. Dissolve 40 g of KMnO₄ in sufficient 10 percent H₂SO₄ to make 1 liter. Prepare and store in glass bottles to prevent degradation.

6.2 **Analysis.** The reagents needed for analysis are listed below:

6.2.1 **Tin (II) Solution.** Prepare fresh daily and keep sealed when not being used. Completely dissolve 20 g of tin (II) chloride (or 25 g of tin (II) sulfate) crystals (Baker Analyzed reagent grade or any other brand that will give a clear solution) in 25 ml of concentrated HCl. Dilute to 250 ml with deionized distilled water. Do not substitute HNO₃, H₂SO₄, or other strong acids for the HCl.

6.2.2 Sodium Chloride—Hydroxylamine Solution. Dissolve 12 g of sodium chloride and 12 g of hydroxylamine sulfate (or 12 g of hydroxylamine hydrochloride) in deionized distilled water and dilute to 100 ml.

6.2.3 Hydrochloric Acid (HCl), 8 N. Dilute 67 ml of concentrated HCl to 100 ml with deionized distilled water (slowly add the HCl to the water).

6.2.4 Nitric Acid, 15 Percent (V/V). Dilute 15 ml of concentrated HNO₃ to 100 ml with deionized distilled water.

6.2.5 Mercury Stock Solution, 1 mg Hg/ml. Prepare and store all mercury standard solutions in borosilicate glass containers. Completely dissolve 0.1354 g of mercury (II) chloride in 75 ml of deionized distilled water. Add 100 ml of concentrated HNO₃, and adjust the volume to exactly 100 ml with deionized distilled water. Mix thoroughly. This solution is stable for at least 1 month.

6.2.6 Intermediate Mercury Standard Solution, 10 µg Hg/ml. Prepare fresh weekly. Pipet 5.0 ml of the mercury stock solution (Section 6.2.5) into a 500-ml volumetric flask and add 20 ml of 15 percent HNO₃ solution. Adjust the volume to exactly 500 ml with deionized distilled water. Thoroughly mix the solution.

6.2.7 Working Mercury Standard Solution, 200 ng Hg/ml. Prepare fresh daily. Pipet 5.0 ml from the "Intermediate Mercury Standard Solution" (Section 6.2.6) into a 250-ml volumetric flask. Add 5 ml of 4 percent KMnO₄ absorbing solution and 5 ml of 15 percent HNO₃. Adjust the volume to exactly 250 ml with deionized distilled water. Mix thoroughly.

6.2.8 Potassium Permanganate, 5 Percent (W/V). Dissolve 5 g of KMnO₄ in deionized distilled water and dilute to 100 ml.

6.2.9 Filter. Whatman No. 40 or equivalent.

7. Procedure

7.1 Sampling. The sampling procedure is the same as Method 101, except for changes due to the use of KMnO₄ instead of ICl absorbing solution and the possible use of a filter. These changes are as follows:

7.1.1 Preliminary Determinations. The preliminary determinations are the same as those given in Method 101, section 7.1.2, except for the absorbing solution depletion sign. In this method, high oxidizable organic content may make it impossible to sample for the desired minimum time. This problem is indicated by the complete bleaching of the purple color of the KMnO₄ solution. In these cases, the tester may divide the sample run into two or more subruns to insure that the absorbing solution would not be depleted. In cases where an excess of water condensation is encountered, collect two runs to make one sample.

7.1.2 Preparation of Sampling Train. The preparation of the sampling train is the same as that given in Method 101, section 7.1.3, except for the cleaning of the glassware [probe, filter holder (if used), impingers, and connectors] and the charging of the first three impingers. In this method, clean all the glass components by rinsing with 50 percent HNO₃, tap water, 8 N HCl, tap water, and finally deionized distilled water. Then place 50 ml of 4 percent KMnO₄ in the first impinger and 100 ml in each of the second and third impingers.

If a filter is used, use a pair of tweezers to place the filter in the filter holder. Be sure to center the filter and place the gasket in proper position to prevent the sample gas stream from by-passing the filter. Check the filter for tears after assembly is completed. Be sure also to set the filter heating system at the desired operating temperature after the sampling train has been assembled.

7.1.3 Sampling Train Operation. In addition to the procedure given in Method 101, section 7.1.5, maintain a temperature around the filter (if applicable) of 120°±14°C (248°±25°F).

7.2 Sample Recovery. Begin proper cleanup procedure as soon as the probe is removed from the stack at the end of the sampling period. Allow the probe to cool. When it can be safely handled, wipe off any external particulate matter near the tip of the probe nozzle and place a cap over it. Do not cap off the probe tip tightly while the sampling train is cooling because the resultant vacuum would draw liquid out from the impingers.

Before moving the sample train to the cleanup site, remove the probe from the train, wipe off the silicone grease, and cap the open outlet of the probe. Be careful not to lose any condensate that might be present. Wipe off the silicone grease from the impinger. Use either ground-glass stoppers, plastic caps, or serum caps to close these openings.

Transfer the probe, impinger assembly, and (if applicable) filter assembly to a cleanup area that is clean, protected from the wind, and free of Hg contamination. The ambient air in laboratories located in the immediate vicinity of Hg-using facilities is not normally free of Hg contamination.

Inspect the train before and during assembly, and note any abnormal conditions. Treat the sample as follows:

7.2.1 Container No. 1 (Impinger, Probe, and Filter Holder). Use a graduated cylinder; measure the liquid in the first three impingers to within ±1 ml. Record the volume of liquid present (e.g., see Figure 5-3 of Method 5 in part 60 of 40 CFR). This information is needed to calculate the moisture content of the effluent gas. (Use only graduated cylinder and glass storage bottles that

have been precleaned as in section 7.1.2.) Place the contents of the first three impingers into a 1000-ml glass sample bottle.

(NOTE: If a filter is used, remove the filter from its holder, as outlined under "Container No. 3" below.)

Taking care that dust on the outside of the probe or other exterior surfaces does not get into the sample, quantitatively recover the Hg (and any condensate) from the probe nozzle, probe fitting, probe liner and front half of the filter holder (if applicable) as follows: Rinse these components with a total of 250 to 400 ml of fresh 4 percent KMnO₄ solution; add all washings to the 1000-ml glass sample bottle; remove any residual brown deposits on the glassware using the minimum amount of 8 N HCl required; and add this HCl rinse to this sample container.

After all washings have been collected in the sample container, tighten the lid on the container to prevent leakage during shipment to the laboratory. Mark the height of the fluid level to determine whether leakage occurs during transport. Label the container to clearly identify its contents.

7.2.2. Container No. 2 (Silica Gel). Note the color of the indicating silica gel to determine whether it has been completely spent and make a notation of its condition. Transfer the silica gel from its impinger to its original container and seal. The tester may use as aids a funnel to pour the silica gel and a rubber policeman to remove the silica gel from the impinger. It is not necessary to remove the small amount of particles that may adhere to the impinger wall and are difficult to remove. Since the gain in weight is to be used for moisture calculations, do not use any water or other liquids to transfer the silica gel. If a balance is available in the field, weigh the spent silica gel (or silica gel plus impinger) to the nearest 0.5 g; record this weight.

7.2.3. Container No. 3 (Filter). If a filter was used, carefully remove it from the filter holder, place it in a 100-ml glass sample bottle, and add 20 to 40 ml of 4 percent KMnO₄. If it is necessary to fold the filter, be sure that the particulate cake is inside the fold. Carefully transfer to the 150-ml sample bottle any particulate matter and filter fibers that adhere to the filter holder gasket by using a dry Nylon bristle brush and a sharp-edged blade. Seal the container. Label the container to clearly identify its contents. Mark the height of the fluid level to determine whether leakage occurs during transport.

7.2.4. Container No. 4 (Filter Blank). If a filter was used, treat an unused filter from the same filter lot used for sampling in the same manner as Container No. 3.

7.2.5. Container No. 5 (Absorbing Solution Blank). For a blank, place 500 ml of 4

percent KMnO₄ absorbing solution in a 1000-ml sample bottle. Seal the container.

7.3 Sample preparation. Check liquid level in each container to see if liquid was lost during transport. If a noticeable amount of leakage occurred, either void the sample or use methods subject to the approval of the Administrator to account for the losses. Then follow the procedures below.

7.3.1. Containers No. 3 and No. 4 (Filter and Filter Blank). If a filter was used, place the contents, including the filter, of Containers No. 3 and No. 4 in separate 250-ml beakers and heat the beakers on a steam bath until most of the liquid has evaporated. Do not take to dryness. Add 20 ml of concentrated HNO₃ to the beakers, cover them with a glass, and heat on a hot plate at 70° C for 2 hours. Remove from the hot plate and filter the solution through Whatman No. 40 filter paper. Save the filtrate for Hg analysis. Discard the filter.

7.3.2. Container No. 1 (Impingers, Probe, and Filter Holder). Filter the contents of Container No. 1 through Whatman 40 filter paper to remove the brown MnO₂ precipitate. Wash the filter with 50 ml of 4 percent KMnO₄ absorbing solution and add this wash to the filtrate. Discard the filter. Combine the filtrates from Containers No. 1 and No. 3 (if applicable), and dilute to a known volume with deionized distilled water. Mix thoroughly.

7.3.3. Container No. 5 (Absorbing Solution Blank). Treat this container as described in section 7.3.2. Combine this filtrate with the filtrate with Container No. 4 and dilute to a known volume with deionized distilled water. Mix thoroughly.

7.4 Analysis. Calibrate the spectrophotometer and recorder and prepare the calibration curve as described in sections 8.1 and 8.2. Then repeat the procedure used to establish the calibration curve with appropriately sized aliquots (1 to 10 ml) of the samples (from sections 7.3.2 and 7.3.3) until two consecutive peak heights agree within ± 3 percent of their average value. If the 10-ml sample is below the detectable limit, use a larger aliquot (up to 20 ml), but decrease the volume of water added to the aeration cell accordingly to prevent the solution volume from exceeding the capacity of the aeration bottle. If the peak maximum of a 1.0-ml aliquot is off scale, further dilute the original sample to bring the Hg concentration into the calibration range of the spectrophotometer. If the Hg content of the absorbing solution and filter blank is below the working range of the analytical method, use zero for the blank.

Run a blank and standard at least after every five samples to check the spectrophotometer calibration; recalibrate as necessary.

It is also recommended that at least one sample from each stack test be checked by the Method of Standard Additions to confirm that matrix effects have not interfered in the analysis.

8. Calibration and Standards

The calibration and standards are the same as Method 101, Section 8, except for the following variations:

8.1 Optical Cell Heating System Calibration. Same as Method 101, Section 8.2, except use a 25-ml graduated cylinder to add 25 ml of deionized distilled water to the bottle section of the aeration cell.

8.2 Spectrophotometer and Recorder Calibration. The mercury response may be measured by either peak height or peak area. (NOTE: The temperature of the solution affects the rate at which elemental Hg is released from a solution and, consequently, it affects the shape of the absorption curve (area) and the point of maximum absorbance (peak height). To obtain reproducible results, all solutions must be brought to room temperature before use.) Set the spectrophotometer wave length at 253.7 nm and make certain the optical cell is at the minimum temperature that will prevent water condensation.

Then set the recorder scale as follows: Using a 25-ml graduated cylinder, add 25 ml of deionized distilled water to the aeration cell bottle and pipet 5.0 ml of the working mercury standard solution into the aeration cell. (NOTE: Always add the Hg-containing solution to the aeration cell after the 25 ml of deionized distilled water.) Place a Teflon-coated stirring bar in the bottle. Add 5 ml of the 4 percent KMnO_4 absorbing solution followed by 5 ml of 15 percent HNO_3 and 5 ml of 5 percent KMnO_4 to the aeration bottle and mix well. Now, attach the bottle section to the bubbler section of the aeration cell and make certain that (1) the aeration cell exit arm stopcock (Figure 101-3 of Method 101) is closed (so that Hg will not prematurely enter the optical cell when the reducing agent is being added) and (2) there is no flow through the bubbler. Add 5 ml of sodium chloride hydroxylamine in 1-ml increments until the solution is colorless. Now add 5 ml of tin (II) solution to the aeration bottle through the side arm, and immediately stopper the side arm. Stir the solution for 15 seconds, turn on the recorder, open the aeration cell exit arm stopcock, and immediately initiate aeration with continued stirring. Determine the maximum absorbance of the standard and set this value to read 90 percent of the recorder full scale.

9. Calculations

9.1 Dry Gas Volume, Volume of Water Vapor and Moisture Content, Stack Gas Velocity, Isokinetic Variation and Acceptable Results, and Determination of Compliance. Same as Method 101, sections 9.1, 9.2, 9.3,

9.6, and 9.7, respectively, except use data obtained from this test.

9.2 Total Mercury. For each source sample, correct the average maximum absorbance of the two consecutive samples whose peak heights agreed within ± 3 percent of their average for the contribution of the field blank. Then calculate the total Hg content in μg in each sample. Correct for any dilutions made to bring the sample into the working range of the spectrophotometer.

9.3 Mercury Emission Rate. Calculate the Hg emission rate R in g/day for continuous operations using Equation 101A-1. For cyclic operations, use only the time per day each stack is in operation. The total Hg emission rate from a source will be the summation of results from all stacks.

$$R = K \frac{m_{\text{Hg}} V_s A_s (86,400 \times 10^{-6})}{[V_{\text{w(sat)}} + V_{\text{w(sat)}}](T_s/P_s)}$$

Eq. 101A-1

Where:

m_{Hg} = Total Hg content in each sample, μg .

V_s = Average stack gas velocity, m/sec (ft/min).

A_s = Stack cross-sectional area, m^2 (ft^2).

86,400 = Conversion factor, sec/day.

10^{-6} = Conversion factor, g/ μg .

$V_{\text{w(sat)}}$ = Dry gas sample volume at standard conditions, corrected for leakage (if any), m^3 (ft^3).

$V_{\text{w(sat)}}$ = Volume of water vapor at standard conditions, m^3 (ft^3).

T_s = Absolute average stack gas temperature, $^{\circ}\text{K}$ ($^{\circ}\text{R}$).

P_s = Absolute stack gas pressure, mm Hg (in. Hg).

$K = 0.3858$ $^{\circ}\text{K}/\text{mm Hg}$ for metric units.

$= 17.64$ $^{\circ}\text{R}/\text{in. Hg}$ for English units.

10. Bibliography

1. Same as Method 101, Section 10.
2. Mitchell, W. J., M. R. Midgett, J. C. Suggs, and D. Albrinck. Test Methods to Determine the Mercury Emissions from Sludge Incineration Plants. U.S. Environmental Protection Agency, Research Triangle Park, North Carolina. Publication No. EPA-600/4-79-058. September 1979.

METHOD 102—DETERMINATION OF PARTICULATE AND GASEOUS MERCURY EMISSIONS FROM CHLOR-ALKALI PLANTS—HYDROGEN STREAMS

1. Introduction

Although similar to Method 101, Method 102 requires changes to accommodate the sample being extracted from a hydrogen

stream. Conduct the test according to Method 101, except as shown below:

2. Mercury Train Operation

2.1 Probe Heating System. Do not use, unless otherwise specified.

2.2 Glass Fiber Filter. Do not use, unless otherwise specified.

2.3 Safety Procedures. The sampler must conduct the source test under conditions of utmost safety, because hydrogen and air mixtures are explosive. Since the sampling train essentially is leakless, attention to safe operation can be concentrated at the inlet and outlet. If a leak does occur, however, remove the meter box cover to avoid a possible explosive mixture. The following specific precautions are recommended:

2.3.1 Operate only the vacuum pump during the test. The other electrical equipment, e.g., heaters, fans, and timers, normally are not essential to the success of a hydrogen stream test.

2.3.2 Seal the sample port to minimize leakage of hydrogen from the stack.

2.3.3 Vent sampled hydrogen at least 3 m (10 feet) away from the train. This can be accomplished by attaching a 13-mm-ID (0.50-in.) Tygon tube to the exhaust from the orifice meter. (NOTE: A smaller ID tubing may cause the orifice meter calibration to be erroneous.) Take care to ensure that the exhaust line is not bent or pinched.

2.4 Setting of Isokinetic Rates.

2.4.1 If a nomograph is used, take special care in the calculation of the molecular weight of the stack gas and in the setting of the nomograph to maintain isokinetic conditions during sampling (sections 2.4.1.1 through 2.4.1.3 below).

2.4.1.1 Calibrate the meter box orifice. Use the techniques described in APTD-0576 (see Citation 9 in section 10 of Method 101). Calibration of the orifice meter at flow conditions that simulate the conditions at the source is suggested. Calibration should either be done with hydrogen or with some other gas having a similar Reynolds Number so that there is similarity between the Reynolds Numbers during calibration and during sampling.

2.4.1.2 The nomograph described in APTD-0576 cannot be used to calculate the C factor because the nomograph is designed for use when the stack gas dry molecular weight is 29 ± 4 . Instead, the following calculation should be made to determine the proper C factor:

$$C = 0.00154 \Delta H_m C_p T_m (P_1/P_m) \frac{(1 - B_m)^2}{(1 - B_m) + 18 B_m / M_d}$$

Where:

ΔH_m = Meter box calibration factor obtained in Section 2.4.1.1, in H_2O .

C_p = Pitot tube calibration coefficient, dimensionless.

T_m = Absolute temperature of gas at the orifice, $^{\circ}R$.

P_1 = Absolute pressure of stack gas, in. Hg.

P_m = Absolute pressure of gas at the meter, in. Hg.

B_m = Fraction by volume of water vapor in the stack gas.

M_d = Dry molecular weight of stack gas, lb/lb-mole.

$0.00154 = (\text{in. } H_2O) (^{\circ}R)$

NOTE. This calculation is left in English units, and is not converted to metric units because nomographs are based on English units.

2.4.1.3 Set the calculated C factor on the operating nomograph and select the proper nozzle diameter and K factor as specified in APTD-0576. If the C factor obtained in Section 2.4.1.2 exceeds the values specified on the existing operating nomograph, expand the C scale logarithmically so that the values can be properly located.

2.4.2 If a calculator is used to set isokinetic rates, it is suggested that the isokinetic equation presented in Citation 17 in the Bibliography of Method 101 be used.

2.5 Sampling in Small (<12-in.-Diameter) Stacks. When the stack diameter (or equivalent diameter) is less than 12 inches, conventional pitot tube-probe assemblies should not be used. For sampling guidelines, see Citation 18 in the Bibliography of Method 101.

METHOD 103—BERYLLIUM SCREENING METHOD

1. Applicability and Principle

1.1 Applicability. This procedure details guidelines and requirements for methods acceptable for use in determining beryllium (Be) emissions in ducts or stacks at stationary sources.

1.2 Principle. Be emissions are isokinetically sampled from three points in a duct or stack. The collected sample is analyzed for Be using an appropriate technique.

2. Apparatus

2.1 Sampling Train. A schematic of the required sampling train configuration is shown in Figure 103-1. The essential components of the train are the following:

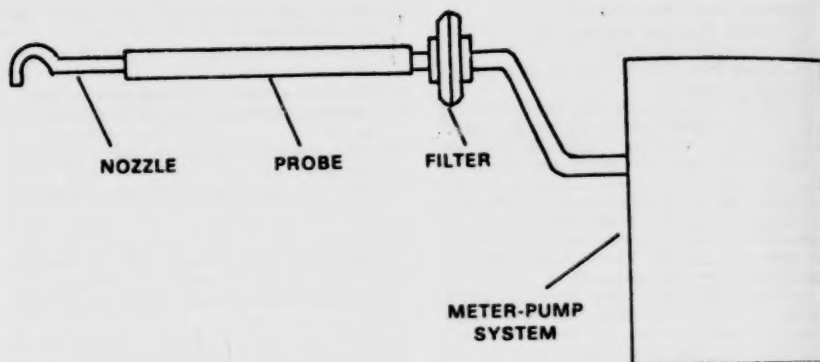


Figure 103-1. Beryllium screening method; sample train schematic.

2.1.1 Nozzle. Stainless steel, or equivalent, with sharp, tapered leading edge.

2.1.2 Probe. Sheathed borosilicate or quartz glass tubing.

2.1.3 Filter. Millipore AA (*Note:* Mention of trade names or specific products does not constitute endorsement by the Environmental Protection Agency), or equivalent, with appropriate filter holder that provides a positive seal against leakage from outside or around the filter. It is suggested that a Whatman 41, or equivalent, be placed immediately against the back side of the Millipore filter as a guard against breakage of the Millipore. Include the backup filter in the analysis. To be equivalent, other filters shall exhibit at least 99.95 percent efficiency (0.05 percent penetration) on 0.3 micron dioctyl phthalate smoke particles, and be amenable to the Be analysis procedure. The filter efficiency tests shall be conducted in accordance with American Society for Testing and Materials (ASTM) Standard Method D 2986-71 (reapproved 1978) (incorporated by reference—see § 61.18). Test data from the supplier's quality control program are sufficient for this purpose.

2.1.4 Meter-Pump System. Any system that will maintain isokinetic sampling rate, determine sample volume, and is capable of a sampling rate of greater than 14 lpm (0.5 cfm).

2.2 Measurement of Stack Conditions. The following equipment is used to measure stack conditions:

2.2.1 Pitot Tube. Type S, or equivalent, with a coefficient within 5 percent over the working range.

2.2.2 Inclined Manometer, or Equivalent. To measure velocity head to within 10 percent of the minimum value.

2.2.3 Temperature Measuring Device. To measure stack temperature to within 1.5 percent of the minimum absolute stack temperature.

2.2.4 Pressure Measuring Device. To measure stack pressure to within 2.5 mm Hg (0.1 in. Hg).

2.2.5 Barometer. To measure atmospheric pressure to within 2.5 mm Hg (0.1 in. Hg).

2.2.6 Wet and Dry Bulb Thermometers, Drying Tubes, Condensers, or Equivalent. To determine stack gas moisture content to within 1 percent.

2.3 Sample Recovery.

2.3.1 Probe Cleaning Equipment. Probe brush or cleaning rod at least as long as probe, or equivalent. Clean cotton balls, or equivalent, should be used with the rod.

2.3.2 Leakless Glass Sample Bottles. To contain sample.

2.4 Analysis. Use equipment necessary to perform an atomic absorption, spectrographic, fluorometric, chromatographic, or equivalent analysis.

3. Reagents

3.1 Sample Recovery.

3.1.1 Water. Distilled water.

3.1.2 Acetone. Reagent grade.

3.1.3 Wash Acid, 50 Percent (V/V) Hydrochloric Acid (HCl).

Mix equal volumes of concentrated HCl and water, being careful to add the acid slowly to the water.

3.2 Analysis. Reagents as necessary for the selected analytical procedure.

4. Procedure

Guidelines for source testing are detailed in the following sections. These guidelines are generally applicable; however, most sample sites differ to some degree and temporary alterations such as stack extensions or expansions often are required to insure the best possible sample site. Further, since Be is hazardous, care should be taken to minimize exposure. Finally, since the total quantity of Be to be collected is quite small, the test must be carefully conducted to prevent contamination or loss of sample.

4.1 Selection of a Sampling Site and Number of Sample Runs. Select a suitable sample site that is as close as practicable to the point of atmospheric emission. If possible, stacks smaller than 1 foot in diameter should not be sampled.

4.1.1 Ideal Sampling Site. The ideal sampling site is at least eight stack or duct diameters downstream and two diameters upstream from any flow disturbance such as a bend, expansion or contraction. For rectangular cross sections, use Equation 103-1 to determine an equivalent diameter, D_e .

$$D_e = \frac{2LW}{L+W} \quad \text{Eq. 103-1}$$

Where:

L = length

W = width

4.1.2 Alternate Sampling Site. Some sampling situations may render the above sampling site criteria impractical. In such cases, select an alternate site no less than two diameters downstream and one-half diameter upstream from any point of flow disturbance. Additional sample runs are recommended at any sample site not meeting the criteria of section 4.1.1.

4.1.3 Number of Sample Runs Per Test. Three sample runs constitute a test. Conduct each run at one of three different points. Select three points that proportionately divide the diameter, or are located at 25, 50, and 75 percent of the diameter from the inside wall. For horizontal ducts, sample on a vertical line through the centroid. For rectangular ducts, sample on a line through the centroid and parallel to a side. If additional sample runs are performed per section 4.1.2, proportionately divide the duct to accommodate the total number of runs.

4.2 Measurement of Stack Conditions. Using the equipment described in section 2.2, measure the stack gas pressure, moisture, and temperature to determine the mo-

lecular weight of the stack gas. Sound engineering estimates may be made in lieu of direct measurements. Describe the basis for such estimates in the test report.

4.3 Preparation of Sampling Train. Assemble the sampling train as shown in Figure 103-1. It is recommended that all glassware be precleaned by soaking in wash acid for 2 hours.

Leak check the sampling train at the sampling site. The leakage rate should not be in excess of 1 percent of the desired sample rate.

4.4 Beryllium Train Operation. For each run, measure the velocity at the selected sampling point. Determine the isokinetic sampling rate. Record the velocity head and the required sampling rate. Place the nozzle at the sampling point with the tip pointing directly into the gas stream. Immediately start the pump and adjust the flow to isokinetic conditions. At the conclusion of the test, record the sampling rate. Again measure the velocity head at the sampling point. The required isokinetic rate at the end of the period should not have deviated more than 20 percent from that originally calculated. Describe the reason for any deviation beyond 20 percent in the test report.

Sample at a minimum rate of 14 lpm (0.5 cfm). Obtain samples over such a period or periods of time as are necessary to determine the maximum emissions which would occur in a 24-hour period. In the case of cyclic operations, perform sufficient sample runs so as to allow determination or calculation of the emissions that occur over the duration of the cycle. A minimum sampling time of 2 hours per run is recommended.

4.5 Sample Recovery. It is recommended that all glassware be precleaned as in section 4.3. Sample recovery should also be performed in an area free of possible Be contamination. When the sampling train is moved, exercise care to prevent breakage and contamination. Set aside a portion of the acetone used in the sample recovery as a blank for analysis. The total amount of acetone used should be measured for accurate blank correction. Blanks can be eliminated if prior analysis shows negligible amounts.

Remove the filter (and backup filter, if used) and any loose particulate matter from filter holder, and place in a container.

Clean the probe with acetone and a brush or long rod and cotton balls. Wash into the container with the filter. Wash out the filter holder with acetone, and add to the same container.

4.6 Analysis. Make the necessary preparation of samples and analyze for Be. Any currently acceptable method such as atomic absorption, spectrographic, fluorometric, chromatographic, or equivalent may be used.

5. Calibration and Standards

5.1 Sampling Train. As a procedural check, compare the sampling rate regulation with a dry gas meter, spirometer, rotameter (calibrated for prevailing atmospheric conditions), or equivalent, attached to the nozzle inlet of the complete sampling train.

5.2 Analysis. Perform the analysis standardization as suggested by the manufacturer of the instrument, or the procedures for the analytical method in use.

6. Calculations

Calculate the Be emission rate R in g/day for each stack using Equation 103-2. For cyclic operations, use only the time per day each stack is in operation. The total Be emission rate from a source is the summation of results from all stacks.

$$R = \frac{W_t(\text{avg}) A_s (86,400 \times 10^{-6})}{V_{\text{total}}}$$

Eq. 103-2

Where:

W_t = Total weight of Be collected, μg .

$v_s(\text{avg})$ = Average stack gas velocity, m/sec (ft/sec).

$A_s(\text{avg})$ = Stack area, $\text{m}^2(\text{ft}^2)$.

86,400 = Conversion factor, sec/day.

10^{-6} = Conversion factor, g/ μg .

V_{total} = Total volume of gas sampled, $\text{m}^3(\text{ft}^3)$.

7. Test Report

Prepare a test report that includes as a minimum: A detailed description of the sampling train used, results of the procedural check described in Section 5.1 with all data and calculations made, all pertinent data taken during the test, the basis for any estimates made, isokinetic sampling calculations, and emission results. Include a description of the test site, with a block diagram and brief description of the process, location of the sample points in the stack cross section, and stack dimensions and distances from any point of disturbance.

[38 FR 8826, Apr. 6, 1973, as amended at 50 FR 46295, Nov. 7, 1985]

METHOD 104—DETERMINATION OF BERYLLIUM EMISSIONS FROM STATIONARY SOURCES

1. Applicability and Principle

1.1 Applicability. This method is applicable for the determination of beryllium (Be) emissions in ducts or stacks at stationary sources. Unless otherwise specified, this method is not intended to apply to gas streams other than those emitted directly to the atmosphere without further processing.

1.2 Principle. Be emissions are isokinetically sampled from the source, and the collected sample is digested in an acid solution and analyzed by atomic absorption spectrophotometry.

2. Apparatus

2.1 Sampling Train. The sampling train is identical to the Method 5 train as shown in Figure 5-1 (mention of Method 5 refers to 40 CFR part 60). The sampling train consists of the following components:

2.1.1 Probe Nozzle, Pitot Tube, Differential Pressure Gauge, Metering System, Barometer, and Gas Density Determination Equipment. Same as Method 5, Sections 2.1.1, 2.1.3, 2.1.4, 2.1.8, 2.1.9, and 2.1.10, respectively.

2.1.2 Probe Liner. Borosilicate or quartz glass tubing. The tester may use a heating system capable of maintaining a gas temperature of $120 \pm 14^\circ\text{C}$ ($248 \pm 25^\circ\text{F}$) at the probe exit during sampling to prevent water condensation. *Note:* Do not use metal probe liners.

2.1.3 Filter Holder. Borosilicate glass, with a glass frit filter support and a silicone rubber gasket. Other materials of construction (e.g., stainless steel, Teflon, Viton) may be used, subject to the approval of the Administrator. (*Note:* Mention of trade names of specific products does not constitute endorsement by the Environmental Protection Agency.) The holder design shall provide a positive seal against leakage from the outside or around the filter. The holder shall be attached immediately at the outlet of the probe. A heating system capable of maintaining the filter at a minimum temperature in the range of the stack temperature may be used to prevent condensation from occurring.

2.1.4 Impingers. Four Greenburg-Smith impingers connected in series with leak-free ground glass fittings or any similar leak-free noncontaminating fittings. For the first, third, and fourth impingers, the tester may use impingers that are modified by replacing the tip with a 13-mm-ID (0.5-in.) glass tube extending to 13 mm (0.5 in.) from the bottom of the flask.

2.2 Sample Recovery. The following items are needed:

2.2.1 Probe Cleaning Rod. At least as long as probe.

2.2.2 Glass Sample Bottles. Leakless, with Teflon-lined caps, 500-ml.

2.2.3 Graduated Cylinder. 250-ml.

2.2.4 Funnel and Rubber Policeman. To aid in transfer of silica gel to container; not necessary if silica gel is weighed in the field.

2.2.5 Funnel. Glass, to aid in sample recovery.

2.2.6 Plastic Jar. Approximately 300-ml.

2.3 Analysis. The following equipment is needed:

2.3.1 Atomic Absorption Spectrophotometer. Perkin-Elmer 303, or equivalent, with nitrous oxide/acetylene burner.

2.3.2 Hot Plate.

2.3.3 Perchloric Acid Fume Hood.

3. Reagents

Use ACS reagent-grade chemicals or equivalent, unless otherwise specified.

3.1 Sampling and Recovery. The reagents used in sampling and recovery are as follows:

3.1.1 Filter. Millipore AA, or equivalent. It is suggested that a Whatman 41 filter or equivalent be placed immediately against the back side of the Millipore filter as a guard against breaking the Millipore filter. To be equivalent, other filters shall exhibit at least 99.95 percent efficiency (0.05 percent penetration) on 0.3 micron dioctyl phthalate smoke particles. The filter efficiency tests shall be conducted in accordance with ASTM Standard Method D 2986-71 (reapproved 1978) (incorporated by reference—see § 61.18). Test data from the supplier's quality control program are sufficient for this purpose.

3.1.2 Water. Deionized distilled, meeting ASTM Specifications for Type 3 Reagent Water—ASTM Test Method D 1193-77 (incorporated by reference—see § 61.18). If high concentrations of organic matter are not expected to be present, the analyst may eliminate the KMnO_4 test for oxidizable organic matter.

3.1.3 Silica Gel. Indicating type, 6- to 16-mesh. If previously used, dry at 175°C (350°F) for 2 hours. The tester may use new silica gel as received.

3.1.4 Acetone.

3.1.5 Wash Acid, 50 Percent (V/V) Hydrochloric Acid (HCl).

Mix equal volumes of concentrated HCl and water, being careful to add the acid slowly to the water.

3.2 Sample Preparation and Analysis. The reagents needed are listed below:

3.2.1 Water. Same as Section 3.1.2.

3.2.2 Perchloric Acid (HClO_4). Concentrated (70 percent).

3.2.3 Nitric Acid (HNO_3). Concentrated.

3.2.4 Beryllium Powder. Minimum purity 98 percent.

3.2.5 Sulfuric Acid (H_2SO_4) Solution, 12 N. Dilute 33 ml of concentrated H_2SO_4 to 1 liter with water.

3.2.6 Hydrochloric Acid Solution, 25 Percent HCl (V/V).

3.2.7 Standard Beryllium Solution, 1 μg Be/ml. Dissolve 10 mg of Be in 80 ml of 12 N H_2SO_4 solution, and dilute to 1000 ml with water. Dilute a 10-ml aliquot to 100 ml with 25 percent HCl solution to give a concentration of 1 μg /ml. Prepare this dilute stock solution fresh daily. Equivalent strength Be stock solutions may be pre-

pared from Be salts such as BeCl_2 and $\text{Be}(\text{NO}_3)_2$ (98 percent minimum purity).

4. Procedure

4.1 Sampling. Because of the complexity of this method testers should be trained and experienced with the test procedures to assure reliable results. As Be is hazardous, testers should take precautions to minimize exposure. The amount of Be that is collected is generally small, therefore, it is necessary to exercise particular care to prevent contamination or loss of sample.

4.1.1 Pretest Preparation. Follow the general procedure given in Method 5, section 4.1.1. Omit the directions for filters, except check them visually against light for irregularities and flaws such as pinholes.

4.1.2 Preliminary Determinations. Follow the general procedure given in Method 5, section 4.1.2, except as follows: Select a nozzle size based on the range of velocity heads to assure that it is not necessary to change the nozzle size in order to maintain isokinetic sampling rates below 28 liters/min (1.0 cfm).

Obtain samples over a period or periods of time that accurately determine the maximum emissions that occur in a 24-hour period. In the case of cyclic operations, perform sufficient sample runs for the accurate determination of the emissions that occur over the duration of the cycle. A minimum sample time of 2 hours per run is recommended.

4.1.3 Prior to assembly, clean all glassware (probe, impingers, and connectors) by first soaking in wash acid for 2 hours, followed by rinsing with water. Place 100 ml of water in each of the first two impingers, and leave the third impinger empty. Save a portion of the water for a blank analysis. Place approximately 200 g of preweighed silica gel in the fourth impinger. The tester may use more silica gel, but should be careful to ensure that it is not entrained and carried out from the impinger during sampling. Place the silica gel container in a clean place for later use in the sample recovery. As an alternative, determine and record the weight of the silica gel plus impinger to the nearest 0.5 g.

Install the selected nozzle using a Viton A O-ring when stack temperatures are less than 260°C (500°F). Use a fiberglass string gasket if temperatures are higher. See APTD-0576 (Citation 9 in Section 10 of Method 101) for details. Other connecting systems using either 316 stainless steel or Teflon ferrules may be used.

If condensation in the probe or filter is a problem, probe and filter heaters will be required. Adjust the heaters to provide a temperature at or above the stack temperature. However, membrane filters such as the Millipore AA are limited to about 225°F. If the

stack gas is in excess of about 200°F, consideration should be given to an alternate procedure such as moving the filter holder downstream of the first impinger to insure that the filter does not exceed its temperature limit. Mark the probe with heat-resistant tape or by some other method to denote the proper distance into the stack or duct for each sampling point. Assemble the train as shown in Figure 5-1 of Method 5, using (if necessary) a very light coat of silicone grease on all ground glass joints. Grease only the outer portion (see APTD-0576) to avoid possibility of contamination by the silicone grease. *Note:* An empty impinger may be inserted between the third impinger and the silica gel to remove excess moisture from the sample stream.

After the sampling train has been assembled, turn on and set the probe, if applicable, at the desired operating temperature. Allow time for the temperatures to stabilize. Place crushed ice around the impingers.

4.1.4. Leak-Check Procedures. Follow the leak-check procedures outlined in Method 5, Sections 4.1.4.1 (Pretest Leak Check), 4.1.4.2 (Leak Checks During Sample Run), and 4.1.4.3 (Post-Test Leak Check).

4.1.5 Beryllium Train Operation. Follow the general procedure given in Method 5, Section 4.1.5. For each run, record the data required on a data sheet such as the one shown in Figure 5-2 of Method 5.

4.1.6 Calculation of Percent Isokinetic. Same as Method 5, Section 4.1.6.

4.2 Sample Recovery. Begin proper cleanup procedure as soon as the probe is removed from the stack at the end of the sampling period.

Allow the probe to cool. When it can be safely handled, wipe off any external particulate matter near the tip of the probe nozzle, and place a cap over it. Do not cap off the probe tip tightly while the sampling train is cooling. Capping would create a vacuum and draw liquid out from the impingers.

Before moving the sampling train to the cleanup site, remove the probe from the train, wipe off the silicone grease, and cap the open outlet of the probe. Be careful not to lose any condensate that might be present. Wipe off the silicone grease from the impinger. Use either ground-glass stoppers, plastic caps, or serum caps to close these openings.

Transfer the probe and impinger assembly to a cleanup area that is clean, protected from the wind, and free of Be contamination.

Inspect the train before and during this assembly, and note any abnormal conditions. Treat the sample as follows:

Disconnect the probe from the impinger train. Remove the filter and any loose particulate matter from the filter holder, and place in a sample bottle. Place the contents

(measured to ± 1 ml) of the first three impingers into another sample bottle. Rinse the probe and all glassware between it and the back half of the third impinger with water and acetone, and add this to the latter sample bottle. Clean the probe with a brush or a long slender rod and cotton balls. Use acetone while cleaning. Add these to the sample bottle. Retain a sample of the water and acetone as a blank. The total amount of water and acetone used should be measured for accurate blank correction. Place the silica gel in the plastic jar. Seal and secure all sample containers for shipment. If an additional test is desired, the glassware can be carefully double rinsed with water and reassembled. However, if the glassware is out of use more than 2 days, repeat the initial acid wash procedure.

4.3 Analysis.

4.3.1 Apparatus Preparation. Before use, clean all glassware according to the procedure of section 4.1.3. Adjust the instrument settings according to the instrument manual, using an absorption wavelength of 234.8 nm.

4.3.2 Sample Preparation. The digestion of Be samples is accomplished in part in concentrated HClO_4 . *Caution:* The analyst must insure that the sample is heated to light brown fumes after the initial HNO_3 addition; otherwise, dangerous perchlorates may result from the subsequent HClO_4 digestion. HClO_4 should be used only under a hood.

4.3.2.1 Filter Preparation. Transfer the filter and any loose particulate matter from the sample container to a 150-ml beaker. Add 35 ml concentrated HNO_3 . Heat on a hotplate until light brown fumes are evident to destroy all organic matter. Cool to room temperature, and add 5 ml concentrated H_2SO_4 and 5 ml concentrated HClO_4 . Then proceed with step 4.3.2.4.

4.3.2.2 Water Preparation. Place a portion of the water and acetone sample into a 150-ml beaker, and put on a hotplate. Add portions of the remainder as evaporation proceeds and evaporate to dryness. Cool the residue, and add 35 ml concentrated HNO_3 . Heat on a hotplate until light brown fumes are evident to destroy any organic matter. Cool to room temperature, and add 5 ml concentrated H_2SO_4 and 5 ml concentrated HClO_4 . Then proceed with step 4.3.2.4.

4.3.2.3 Silica Gel Preparation Analyses. Weigh the spent silica gel, and report to the nearest gram.

4.3.2.4 Final Sample Preparation. Samples from 4.3.2.1 and 4.3.2.2 may be combined here for ease of analysis. Replace on a hotplate, and evaporate to dryness in a HClO_4 hood. Cool and dissolve the residue in 10.0 ml of 25 percent V/V HCl . Samples are now ready for the atomic absorption unit. It is necessary for the Be concentra-

tion of the sample to be within the calibration range of the unit. If necessary, perform further dilution of sample with 25 percent V/V HCl to bring the sample within the calibration range.

4.3.3 Beryllium Determination. Analyze the samples prepared in 4.3.2 at 234.8 nm using a nitrous oxide/acetylene flame. Aluminum, silicon and other elements can interfere with this method if present in large quantities. Standard methods are available, however, that may be used to effectively eliminate these interferences (see Citation 2 in Section 8).

5. Calibration

5.1 Sampling Train. Calibrate the sampling train components according to the procedures outlined in the following sections of Method 5: Section 5.1 (Probe Nozzle), section 5.2 (Pitot Tube), section 5.3 (Metering System), section 5.4 (Probe Heater), section 5.5 (Temperature Gauges), section 5.7 (Barometer). Note that the leak check described in section 5.6 of Method 5 applies to this method.

6. Calculations

6.1 Dry Gas Volume. Using the data from each sample run, calculate the dry gas sample volume at standard conditions $V_{m(Std)}$ (corrected for leakage, if necessary) as outlined in Section 6.3 of Method 5.

6.2 Volume of Water Vapor in Sample and Moisture Content of Stack Gas. Using the data obtained from each sample run, calculate the volume of water vapor $V_{w(Std)}$ in the sample, and the moisture content B_{wv} of the stack gas. Use Equations 5-2 and 5-3 of Method 5.

6.3 Stack Gas Velocity. Using the data from each sample run and Equation 2-9 of Method 2, calculate the average stack gas velocity V_{Avg} .

6.4 Beryllium Emission Rate. Calculate the Be emission rate R in g/day for each stack using Equation 104-1. For cyclic operations, use only the time per day each stack is in operation. The total Be emission rate from a source will be the summation of results from all stacks.

$$R = K \frac{W_i V_{Avg} A_s (86,400 \times 10^{-6})}{[V_{m(Std)} + V_{w(Std)}] (T_s/P_s)}$$

Eq. 104-1

Where:

W_i = Total weight of Be collected, μg .

A_s = Stack cross-sectional area, m^2 (ft^2).

86,400 = Conversion factor, sec/day.

10^{-6} = Conversion factor, g/ μg .

T_s = Absolute average stack gas temperature, $^{\circ}\text{K}$ ($^{\circ}\text{F}$).

P_s = Absolute stack gas pressure, mm Hg (in. Hg).

$K = 0.3858$ $^{\circ}\text{K}/\text{mm Hg}$ for metric units.

$= 17.64$ $^{\circ}\text{P}/\text{in. Hg}$ for English units.

6.5 Isokinetic Variation and Acceptable Results. Same as Method 5, Sections 6.11 and 6.12, respectively.

7. Determination of Compliance

Each performance test consists of three sample runs of the applicable test method. For the purpose of determining compliance with an applicable national emission standard, use the average of the results of all sample runs.

8. Bibliography

In addition to Citations 1-3 and 5-15 of Section 10 of Method 101, the following citations may be helpful:

1. Amos, M.D., and J. B. Willis. Use of High-Temperature Pre-Mixed Flames in Atomic Absorption Spectroscopy. *Spectrochim. Acta* 22:1325. 1966.

2. Fleet, B., K. V. Liberty, and T. S. West. A Study of Some Matrix Effects in the Determination of Beryllium by Atomic Absorption Spectroscopy in the Nitrous Oxide-Acetylene Flame. *Talanta* 17:203. 1970.

METHOD 105—DETERMINATION OF MERCURY IN WASTEWATER TREATMENT PLANT SEWAGE SLUDGE

1. Applicability and Principle

1.1 Applicability. This method applies to the determination of total organic and inorganic mercury (Hg) content in sewage sludges. The range of this method is 0.2 to 5 $\mu\text{g/g}$; it may be extended by increasing or decreasing sample size.

1.2 Principle. Time-composite sludge samples are withdrawn from the conveyor belt after dewatering and before incineration or drying. A weighed portion of the sludge is digested in aqua regia and oxidized by potassium permanganate (KMnO_4). Hg in the digested sample is then measured by the conventional spectrophotometric cold-vapor technique.

2. Apparatus

2.1 Sampling.

2.1.1 Container. Plastic, 50-liter.

2.1.2 Scoop. To remove 950-ml (1-qt.) sludge sample.

2.2 Sludge Sample Preparation.

2.2.1 Mixer. Mortar mixer, wheelbarrow-type, 57-liter (or equivalent) with electricity driven motor.

2.2.2 Blender. Waring-type, 2-liter. (Note: Mention of specific trade names does not constitute endorsement by the Environmental Protection Agency.)

2.2.3 Scoop. To remove 100-ml and 20-ml samples of blended sludge.

2.3 Analysis. Same as Method 101, Sections 5.3 and 5.4, except for the following:

2.3.1 Balance. The balance of Method 101, Section 5.3.17, is not needed.

2.3.2 Filter Paper. S and S No. 588 (or equivalent).

3. Reagents

3.1 Water. Same as Method 101A, Section 6.1.1.

3.2 Aqua Regia. Prepare immediately before use. Carefully add one volume of concentrated nitric acid (HNO_3) to three volumes of concentrated hydrochloric acid (HCl).

3.3 Antifoam B Silicon Emulsion. J.T. Baker Company (or equivalent).

3.4 Mercury (II) Stock Solution, 1 mg Hg/ml. Completely dissolve 135.4 mg of ACS reagent-grade HgCl_2 in 75 ml of water, add 10 ml of concentrated HNO_3 , and adjust the volume to 100.0 ml with water. Mix thoroughly. (This solution is stable for at least 1 month.)

3.5 Intermediate Mercury Standard Solution, 10 μg Hg/ml. Prepare fresh weekly. Pipet 5.0 ml of the Hg stock solution into a 500-ml volumetric flask, and add 20 ml of the 15-percent HNO_3 solution. Adjust the volume to 500 ml with water. Thoroughly mix the solution.

3.6 Working Mercury Standard Solution, 200 ng Hg/ml. Prepare fresh daily. Pipet 5.0 ml of the "Intermediate Mercury Standard Solution" into a 250-ml volumetric flask. Add 20 ml of 15-percent HNO_3 , and adjust the volume to 250 ml with water. Mix thoroughly.

3.7 Tin (II) Solution, Sodium Chloride-Hydroxylamine Solution, 15-Percent Nitric Acid, and Potassium Permanganate Solution. Same as Method 101A, Section 6.2.

4. Procedure

4.1 Sludge Sampling. Withdraw equal-volume increments of sludge [for a total of at least 15 liters (16-qt.)] at intervals of 30 min over an 8-hr period, and place in a rigid plastic container.

4.2 Sludge Mixing. Transfer the entire 15-liter sample to a 57-liter capacity (2-ft³) mortar mixer. Mix the sample for a minimum of 30 min at 30 rpm. Using a 200-ml beaker, take six 100-ml portions of sludge, and combine in a 2-liter blender. Blend sludge for 5 min; add water as necessary to give a fluid consistency. Immediately after stopping the blender, use a 50-ml beaker to withdraw four 20-ml portions of blended sludge, and place them in separate, tared 125-ml Erlenmeyer flasks. Reweigh each flask to determine the exact amount of sludge added. (Use three of the samples to determine the mercury content in the sludge, and use the fourth to measure the solids content of the blended sludge.)

4.3 Solids Content of Blended Sludge. Dry one of the 20-ml blended samples from Section 4.2 in an oven at 105 °C to constant

weight. Cool in a desiccator, and weigh and record the dry weight of the sample.

4.4 Aqua Regia Digestion of Blended Samples. To each of the three remaining 20-ml samples from Section 4.2, add 25 ml of aqua regia, and digest the samples on a hot plate at low heat (do not boil) for 30 min, or until samples are a pale yellow-brown color and are void of the dark brown color characteristic of organic matter. Remove from the hot plate, and allow to cool.

Filter each digested sample separately through an S and S No. 588 filter, or equivalent, and rinse the filter contents with 50 ml of water. Transfer the filtrate and filter washing to a 100-ml volumetric flask, and carefully dilute to volume with water.

4.5 Solids Content of Sludge Before Blending. Using a 200-ml beaker, remove two 100-ml portions of mixed sludge from the mortar mixer, and place in separate, tared 400-ml beakers. Reweigh each beaker to determine the exact amount of sludge added. Dry in an oven at 105 °C, and cool in a desiccator to constant weight.

4.6 Analysis for Mercury. The same as Method 101A, Sections 7.4 and 8, except for the following variation.

4.6.1 Spectrophotometer and Recorder Calibration. The mercury response may be measured by either peak height or peak area. Note: The temperature of the solution affects the rate at which elemental Hg is released from solution and, consequently, it affects the shape of the absorption curve (area) and the point of maximum absorbance (peak height). Therefore, to obtain reproducible results, bring all solutions to room temperature before use.

Set the spectrophotometer wavelength to 253.7 nm. Make certain the optical cell is at the minimum temperature that will prevent water condensation from occurring. Then set the recorder scale as follows: Using a 25-ml graduated cylinder, add 25 ml of water to the aeration-cell bottle. Add three drops of Antifoam B to the bottle, and then pipet 5.0 ml of the working Hg standard solution into the aeration cell.

NOTE: Always add the Hg containing solution to the aeration cell after the 25 ml of water.

Place a Teflon-coated stirring bar in the bottle. Add 5 ml of 15-percent HNO_3 and 5 ml of 5-percent KMnO_4 to the aeration bottle, and mix well. Next, attach the bottle section to the bubbler section of the aeration cell, and make certain that: (1) the exit arm stopcock of the aeration cell (Figure 105-3) is closed (so that Hg will not prematurely enter the optical cell when the reducing agent is being added), and (2) there is no flow through the bubbler. Add 5 ml of sodium chloride-hydroxylamine solution to the aeration bottle through the side

arm, and mix. If the solution does not become colorless, add additional sodium chloride-hydroxylamine solution in 1-ml increments until the solution is colorless. Now add 5 ml of tin (II) solution to the aeration bottle through the side arm, and immediately stopper the side arm. Stir the solution for 15 sec, turn on the recorder, open the aeration cell exit arm stopcock, and then immediately initiate aeration with continued stirring. Determine the maximum absorbance of the standard, and set this value to read 90 percent of the recorder full scale.

5. Calculations

5.1 Nomenclature.

C_m = Concentration of Hg in the digested sample, $\mu\text{g/g}$.

F_{ab} = Weight fraction of solids in the blended sludge.

F_{sm} = Weight fraction of solids in the collected sludge after mixing.

M = Hg content of the sewage sludge (on a dry basis), $\mu\text{g/g}$.

m = Mass of Hg in the aliquot of digested sample analyzed, μg .

V_a = Volume of digested sample analyzed, ml.

V_t = Volume of digested sample, ml.

W_t = Weight of empty sample flask, g.

W_b = Weight of sample flask and sample, g.

W_{fb} = Weight of sample flask and sample after drying, g.

W_s = Weight of empty sample beaker, g.

W_{sb} = Weight of sample beaker and sample, g.

W_{mb} = Weight of sample beaker and sample after drying, g.

5.2 Mercury Content of Digested Sample (Wet Basis). For each sample, correct the average maximum absorbance of the two consecutive samples whose peak heights agree within ± 3 percent of their average for the contribution of the blank. Use the calibration curve and these corrected averages to determine the final Hg concentration in the solution cell for each sludge sample.

Calculate the total Hg content in each gram of digested sample correcting for any dilutions made to bring the sample into the working range of the spectrophotometer and for the weight of the sludge portion digested.

$$C_m = \frac{mV_s}{V_s(W_{fb} - W_t)} \quad \text{Eq. 105-1}$$

5.3 Solids Content of Blended Sludge. Determine the solids content of the 20-ml aliquot dried in the oven at 105°C (Section 4.3).

$$F_{ab} = 1 - \frac{W_{fb} - W_{td}}{W_{fb} - W_t} \quad \text{Eq. 105-2}$$

5.4 Solids Content of Bulk Sample (before blending but, after mixing in mortar mixer). Determine the solids content of each 100-ml aliquot (Section 4.5), and average the results.

$$F_{sm} = 1 - \frac{W_{bs} - W_{bd}}{W_{bs} - W_b} \quad \text{Eq. 105-3}$$

5.5 Mercury Content of Bulk Sample (Dry Basis). Average the results from the three samples from each 8-hr composite sample, and calculate the Hg concentration of the composite sample on a dry basis.

$$M = \frac{C_m(\text{avg})}{F_{ab}} \quad \text{Eq. 105-4}$$

6. Bibliography

1. Bishop, J.N. Mercury in Sediments, Ontario Water Resources Commission. Toronto, Ontario, Canada. 1971.
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3. Hatch, W.R. and W.L. Ott. Determination of Sub-Microgram Quantities of Mercury by Atomic Absorption Spectrophotometry. *Analytical Chemistry*. 40:2085. 1968.
4. Bradenberger, H. and H. Bader. The Determination of Nanogram Levels of Mercury in Solution by a Flameless Atomic Absorption Technique. *Atomic Absorption Newsletter*. 6:101. 1967.
5. Analytical Quality Control Laboratory (AQCL). Mercury in Sediment (Cold Vapor Technique) (Provisional Method). U.S. Environmental Protection Agency. Cincinnati, Ohio. April 1972.
6. Kopp, J.F., M.C. Longbottom, and L.B. Lobring. "Cold Vapor" Method for Determining Mercury. *Journal AWWA*. 64(1):20-25. 1972.
7. Manual of Methods for Chemical Analysis of Water and Wastes. U.S. Environmental Protection Agency. Cincinnati, Ohio. Publication No. EPA-624/2-74-003. December 1974. p. 118-138.
8. Mitchell, W.J., M.R. Midgett, J. Suggs, R.J. Velton, and D. Albrinck. Sampling and Homogenizing Sewage for Analysis. Environmental Monitoring and Support Laboratory.

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METHOD 106—DETERMINATION OF VINYL CHLORIDE FROM STATIONARY SOURCES

Introduction

Performance of this method should not be attempted by persons unfamiliar with the operation of a gas chromatograph (GC) nor by those who are unfamiliar with source sampling, because knowledge beyond the scope of this presentation is required. Care must be exercised to prevent exposure of sampling personnel to vinyl chloride, a carcinogen.

1. Applicability and Principle

1.1 **Applicability.** The method is applicable to the measurement of vinyl chloride in stack gases from ethylene dichloride, vinyl chloride, and polyvinyl chloride manufacturing processes. The method does not measure vinyl chloride contained in particulate matter.

1.2 **Principle.** An integrated bag sample of stack gas containing vinyl chloride (chloroethene) is subjected to GC analysis using a flame ionization detector (FID).

2. Range and Sensitivity

This method is designed for the 0.1 to 50 ppm range. However, common GC instruments are capable of detecting 0.02 ppm vinyl chloride. With proper calibration, the upper limit may be extended as needed.

3. Interferences

The chromatographic columns and the corresponding operating parameters herein described normally provide an adequate resolution of vinyl chloride; however, resolution interferences may be encountered on some sources. Therefore, the chromatograph operator shall select the column and operating parameters best suited to his particular analysis requirements, subject to the approval of the Administrator. Approval is automatic, provided that the tester produces confirming data through an adequate supplemental analytical technique, such as analysis with a different column or GC/mass spectroscopy, and has the data available for review by the Administrator.

4. Apparatus

4.1 **Sampling** (see Figure 106-1). The sampling train consists of the following components:

4.1.1 **Probe.** Stainless steel, Pyrex glass, or Teflon tubing (as stack temperature permits) equipped with a glass wool plug to remove particulate matter.

4.1.2 **Sample Lines.** Teflon, 6.4-mm outside diameter, of sufficient length to connect probe to bag. Use a new unused piece for each series of bag samples that consti-

tutes an emission test, and discard upon completion of the test.

4.1.3 **Quick Connects.** Stainless steel, male (2) and female (2), with ball checks (one pair without), located as shown in Figure 106-1.

4.1.4 **Tedlar Bags.** 50- to 100-liter capacity, to contain sample. Aluminized Mylar bags may be used if the samples are analyzed within 24 hours of collection.

4.1.5 **Bag Containers.** Rigid leak-proof containers for sample bags, with covering to protect contents from sunlight.

4.1.6 **Needle Valve.** To adjust sample flow rates.

4.1.7 **Pump.** Leak-free, with minimum of 2-liter/min capacity.

4.1.8 **Charcoal Tube.** To prevent admission of vinyl chloride and other organics to the atmosphere in the vicinity of samplers.

4.1.9 **Flowmeter.** For observing sampling flow rate; capable of measuring a flow range from 0.10 to 1.00 liter/min.

4.1.10 **Connecting Tubing.** Teflon, 6.4-mm outside diameter, to assemble sampling train (Figure 106-1).

4.1.11 **Tubing Fittings and Connectors.** Teflon or stainless steel, to assemble sampling train.

4.2 **Sample Recovery.** Teflon tubing, 6.4-mm outside diameter, to connect bag to GC sample loop for sample recovery. Use a new unused piece for each series of bag samples that constitutes an emission test, and discard upon conclusion of analysis of those bags.

4.3 **Analysis.** The following equipment is required:

4.3.1 **Gas Chromatograph.** With FID, potentiometric strip chart recorder and 1.0- to 5.0-ml heated sampling loop in automatic sample valve. The chromatographic system shall be capable of producing a response to 0.1-ppm vinyl chloride that is at least as great as the average noise level. (Response is measured from the average value of the base line to the maximum of the wave form, while standard operating conditions are in use.)

4.3.2 **Chromatographic Columns.** Columns as listed below. The analyst may use other columns provided that the precision and accuracy of the analysis of vinyl chloride standards are not impaired and he has available for review information confirming that there is adequate resolution of the vinyl chloride peak. (Adequate resolution is defined as an area overlap of not more than 10 percent of the vinyl chloride peak by an interferent peak. Calculation of area overlap is explained in Appendix C, Procedure 1: "Determination of Adequate Chromatographic Peak Resolution.")

4.3.2.1 **Column A.** Stainless steel, 2.0 m by 3.2 mm, containing 80/100-mesh Chromasorb 102.

4.3.2.2 Column B. Stainless steel, 2.0 m by 3.2 mm, containing 20 percent GE SF-96 on 60/80-mesh Chromasorb P AW; or stainless steel, 1.0 m by 3.2 mm containing 80/100-mesh Porapak T. Column B is required as a secondary column if acetaldehyde is present. If used, column B is placed after column A. The combined columns should be operated at 120° C.

4.3.3 Flowmeters (2). Rotameter type, 100-ml/min capacity, with flow control valves.

4.3.4 Gas Regulators. For required gas cylinders.

4.3.5 Thermometer. Accurate to 1° C, to measure temperature of heated sample loop at time of sample injection.

4.3.6 Barometer. Accurate to 5 mm Hg, to measure atmospheric pressure around GC during sample analysis.

4.3.7 Pump. Leak-free, with minimum of 100-ml/min capacity.

4.3.8 Recorder. Strip chart type, optionally equipped with either disc or electronic integrator.

4.3.9 Planimeter. Optional, in place of disc or electronic integrator on recorder, to measure chromatograph peak areas.

4.4 Calibration. Sections 4.4.2 through 4.4.4 are for the optional procedure in Section 7.1.

4.4.1 Tubing. Teflon, 6.4-mm outside diameter, separate pieces marked for each calibration concentration.

4.4.2 Tedlar Bags. Sixteen-inch-square size, with valve; separate bag marked for each calibration concentration.

4.4.3 Syringes. 0.5-ml and 50- μ l, gas tight, individually calibrated to dispense gaseous vinyl chloride.

4.4.4 Dry Gas Meter, with Temperature and Pressure Gauges. Singer Model DTM-115 with 802 index, or equivalent, to meter nitrogen in preparation of standard gas mixtures, calibrated at the flow rate used to prepare standards.

5. Reagents

Use only reagents that are of chromatograph grade.

5.1 Analysis. The following are required for analysis.

5.1.1 Helium or Nitrogen. Zero grade, for chromatographic carrier gas.

5.1.2 Hydrogen. Zero grade.

5.1.3 Oxygen or Air. Zero grade, as required by the detector.

5.2 Calibration. Use one of the following options: either 5.2.1 and 5.2.2, or 5.2.3.

5.2.1 Vinyl Chloride. Pure vinyl chloride gas certified by the manufacturer to contain a minimum of 99.9 percent vinyl chloride, for use in the preparation of standard gas mixtures in Section 7.1. If the gas manufacturer maintains a bulk cylinder supply of 99.9+ percent vinyl chloride, the certification analysis may have been performed on

this supply rather than on each gas cylinder prepared from this bulk supply. The date of gas cylinder preparation and the certified analysis must have been affixed to the cylinder before shipment from the gas manufacturer to the buyer.

5.2.2 Nitrogen. Zero grade, for preparation of standard gas mixtures as described in Section 7.1.

5.2.3 Cylinder Standards (3). Gas mixture standards (50-, 10-, and 5-ppm vinyl chloride in nitrogen cylinders). The tester may use cylinder standards to directly prepare a chromatograph calibration curve as described in Section 7.2.2, if the following conditions are met: (a) The manufacturer certifies the gas composition with an accuracy of ± 3 percent or better (see Section 5.2.3.1). (b) The manufacturer recommends a maximum shelf life over which the gas concentration does not change by greater than ± 5 percent from the certified value. (c) The manufacturer affixes the date of gas cylinder preparation, certified vinyl chloride concentration, and recommended maximum shelf life to the cylinder before shipment to the buyer.

5.2.3.1 Cylinder Standards Certification. The manufacturer shall certify the concentration of vinyl chloride in nitrogen in each cylinder by (a) directly analyzing each cylinder and (b) calibrating his analytical procedure on the day of cylinder analysis. To calibrate his analytical procedure, the manufacturer shall use, as a minimum, a three-point calibration curve. It is recommended that the manufacturer maintain (1) a high-concentration calibration standard (between 50 and 100 ppm) to prepare his calibration curve by an appropriate dilution technique and (2) a low-concentration calibration standard (between 5 and 10 ppm) to verify the dilution technique used. If the difference between the apparent concentration read from the calibration curve and the true concentration assigned to the low-concentration calibration standard exceeds 5 percent of the true concentration, the manufacturer shall determine the source of error and correct it, then repeat the three-point calibration.

5.2.3.2 Verification of Manufacturer's Calibration Standards. Before using a standard, the manufacturer shall verify each calibration standard (a) by comparing it to gas mixtures prepared (with 99 mole percent vinyl chloride) in accordance with the procedure described in Section 7.1 or (b) calibrating it against vinyl chloride cylinder Standard Reference Materials (SRM's) prepared by the National Bureau of Standards, if such SRM's are available. The agreement between the initially determined concentration value and the verification concentration value must be within ± 5 percent. The manufacturer must reverify all calibration

standards on a time interval consistent with the shelf life of the cylinder standards sold.

5.2.4 Audit Cylinder Standards (2). Gas mixture standards with concentrations known only to the person supervising the analysis of samples. The audit cylinder standards shall be identically prepared as those in Section 5.2.3 (vinyl chloride in nitrogen cylinders). The concentrations of the audit cylinder should be: one low-concentration cylinder in the range of 5 to 20 ppm vinyl chloride and one high-concentration cylinder in the range of 20 to 50 ppm. When available, the tester may obtain audit cylinders by contacting: Environmental Protection Agency, Environmental Monitoring Systems Laboratory, Quality Assurance Division (MD-77), Research Triangle Park, North Carolina 27711. Audit cylinders obtained from a commercial gas manufacturer may be used provided: (a) the gas manufacturer certifies the audit cylinder as described in Section 5.2.3.1, and (b) the gas manufacturer obtains an independent analysis of the audit cylinders to verify this analysis. Independent analysis is defined here to mean analysis performed by an individual different than the individual who performs the gas manufacturer's analysis, while using calibration standards and analysis equipment different from those used for the gas manufacturer's analysis. Verification is complete and acceptable when the independent analysis concentration is within ± 5 percent of the gas manufacturer's concentration.

6. Procedure

6.1 Sampling. Assemble the sample train as shown in Figure 106-1. A bag leak check should have been performed previously according to Section 7.3.2. Join the quick connects as illustrated, and determine that all connection between the bag and the probe are tight. Place the end of the probe at the centroid of the stack and start the pump with the needle valve adjusted to yield a flow that will fill over 50 percent of bag volume in the specific sample period. After allowing sufficient time to purge the line several times, change the vacuum line from the container to the bag and evacuate the bag until the rotameter indicates no flow. Then reposition the sample and vacuum lines and begin the actual sampling, keeping the rate proportional to the stack velocity. At all times, direct the gas exiting the rotameter away from sampling personnel. At the end of the sample period, shut off the pump, disconnect the sample line from the bag, and disconnect the vacuum line from the bag container. Protect the bag container from sunlight.

6.2 Sample Storage. Keep the sample bags out of direct sunlight. When at all possible, analysis is to be performed within 24 hours, but in no case in excess of 72 hours

of sample collection. Aluminized Mylar bag samples must be analyzed within 24 hours.

6.3 Sample Recovery. With a new piece of Teflon tubing identified for that bag, connect a bag inlet valve to the gas chromatograph sample valve. Switch the valve to receive gas from the bag through the sample loop. Arrange the equipment so the sample gas passes from the sample valve to 100-ml/min rotameter with flow control valve followed by a charcoal tube and a 1-in. H₂O pressure gauge. The tester may maintain the sample flow either by a vacuum pump or container pressurization if the collection bag remains in the rigid container. After sample loop purging is ceased, allow the pressure gauge to return to zero before activating the gas sampling valve.

6.4 Analysis. Set the column temperature to 100° C and the detector temperature to 150° C. When optimum hydrogen and oxygen flow rates have been determined, verify and maintain these flow rates during all chromatography operations. Using zero helium or nitrogen as the carrier gas, establish a flow rate in the range consistent with the manufacturer's requirements for satisfactory detector operation. A flow rate of approximately 40 ml/min should produce adequate separations. Observe the base line periodically and determine that the noise level has stabilized and that base line drift has ceased. Purge the sample loop for 30 seconds at the rate of 100 ml/min, shut off flow, allow the sample loop pressure to reach atmospheric pressure as indicated by the H₂O manometer, then activate the sample valve. Record the injection time (the position of the pen on the chart at the time of sample injection), sample number, sample loop temperature, column temperature, carrier gas flow rate, chart speed, and attenuator setting. Record the barometric pressure. From the chart, note the peak having the retention time corresponding to vinyl chloride as determined in Section 7.2.1. Measure the vinyl chloride peak area, A_m , by use of a disc integrator, electronic integrator, or a planimeter. Measure and record the peak heights, H_m . Record A_m and retention time. Repeat the injection at least two times or until two consecutive values for the total area of the vinyl chloride peak do not vary more than 5 percent. Use the average value for these two total areas to compute the bag concentration.

Compare the ratio of H_m to A_m for the vinyl chloride sample with the same ratio for the standard peak that is closest in height. If these ratios differ by more than 10 percent, the vinyl chloride peak may not be pure (possibly acetaldehyde is present) and the secondary column should be employed (see Section 4.3.2.2).

6.5 Determination of Bag Water Vapor Content. Measure the ambient temperature

and barometric pressure near the bag. From a water saturation vapor pressure table, determine and record the water vapor content of the bag as a decimal figure. (Assume the relative humidity to be 100 percent unless a lesser value is known.)

7. Preparation of Standard Gas Mixtures, Calibration, and Quality Assurance

7.1 Preparation of Vinyl Chloride Standard Gas Mixtures. (Optional Procedure—delete if cylinder standards are used.) Evacuate a 16-inch square Tedlar bag that has passed a leak check (described in Section 7.3.2) and meter in 5.0 liters of nitrogen. While the bag is filling, use the 0.5-ml syringe to inject 250 μ l of 99.9+ percent vinyl chloride gas through the wall of the bag. Upon withdrawing the syringe, immediately cover the resulting hole with a piece of adhesive tape. The bag now contains a vinyl chloride concentration of 50 ppm. In a like manner use the 50 μ l syringe to prepare gas mixtures having 10- and 5-ppm vinyl chloride concentrations. Place each bag on a smooth surface and alternately depress opposite sides of the bag 50 times to further mix the gases. These gas mixture standards may be used for 10 days from the date of preparation, after which time new gas mixtures must be prepared. (Caution: Contamination may be a problem when a bag is reused if the new gas mixture standard is a lower concentration than the previous gas mixture standard.)

7.2 Calibration.

7.2.1 Determination of Vinyl Chloride Retention Time. (This section can be performed simultaneously with Section 7.2.2.) Establish chromatograph conditions identical with those in Section 6.4 above. Determine proper attenuator position. Flush the sampling loop with zero helium or nitrogen and activate the sample valve. Record the injection time, sample loop temperature, column temperature, carrier gas flow rate, chart speed, and attenuator setting. Record peaks and detector responses that occur in the absence of vinyl chloride. Maintain conditions with the equipment plumbing arranged identically to Section 6.3, and flush the sample loop for 30 seconds at the rate of 100 ml/min with one of the vinyl chloride calibration mixtures. Then activate the sample valve. Record the injection time. Select the peak that corresponds to vinyl chloride. Measure the distance on the chart from the injection time to the time at which the peak maximum occurs. This quantity divided by the chart speed is defined as the retention time. Since other organics may be present in the sample, positive identification of the vinyl chloride peak must be made.

7.2.2 Preparation of Chromatograph Calibration Curve. Make a GC measurement of each gas mixture standard (described in Section 5.2.3 or 7.1) using conditions identi-

cal with those listed in Sections 6.3 and 6.4. Flush the sampling loop for 30 seconds at the rate of 100 ml/min with one of the standard mixtures, and activate the sample valve. Record the concentration of vinyl chloride injected (C_c), attenuator setting, chart speed, peak area, sample loop temperature, column temperature, carrier gas flow rate, and retention time. Record the barometric pressure. Calculate A_c , the peak area multiplied by the attenuator setting. Repeat until two consecutive injection areas are within 5 percent, then plot the average of those two values versus C_c . When the other standard gas mixtures have been similarly analyzed and plotted, draw a straight line through the points derived by the least squares method. Perform calibration daily, or before and after the analysis of each emission test set of bag samples, whichever is more frequent. For each group of sample analyses, use the average of the two calibration curves which bracket that group to determine the respective sample concentrations. If the two calibration curves differ by more than 5 percent from their mean value, then report the final results by both calibration curves.

7.3 Quality Assurance.

7.3.1 Analysis Audit. Immediately after the preparation of the calibration curve and prior to the sample analyses, perform the analysis audit described in Appendix C, Procedure 2: "Procedure for Field Auditing GC Analysis."

7.3.2 Bag Leak Checks. Checking of bags for leaks is required after bag use and strongly recommended before bag use. After each use, connect a water manometer and pressurize the bag to 5 to 10 cm H_2O (2 to 4 in. H_2O). Allow to stand for 10 min. Any displacement in the water manometer indicates a leak. Also, check the rigid container for leaks in this manner. (Note: An alternative leak check method is to pressurize the bag to 5 to 10 cm H_2O and allow it to stand overnight. A deflated bag indicates a leak.) For each sample bag in its rigid container, place a rotameter in line between the bag and the pump inlet. Evacuate the bag. Failure of the rotameter to register zero flow when the bag appears to be empty indicates a leak.

8. Calculations

8.1 Sample Peak Area. Determine the sample peak area, A_s , as follows:

$$A_s = A_m A_t \quad \text{Eq. 106-1}$$

Where:

A_m = Measured peak area.

A_t = Attenuation factor.

8.2 Vinyl Chloride Concentrations. From the calibration curves described in Section 7.2.2, determine the average concentration value of vinyl chloride, C_c , that corresponds to A_c , the sample peak area. Calculate the concentration of vinyl chloride in the bag, C_b , as follows:

$$C_b = \frac{C_c P_r T_i}{P_i T_r (1 - B_{wv})} \quad \text{Eq. 106-2}$$

Where:

C_b = Concentration of vinyl chloride in the bag, ppm.

C_c = Concentration of vinyl chloride in the standard sample, ppm.

P_r = Reference pressure, the laboratory pressure recorded during calibration, mm Hg.

T_i = Sample loop temperature on the absolute scale at the time of analysis, °K.

P_i = Laboratory pressure at time of analysis, mm Hg.

T_r = Reference temperature, the sample loop temperature recorded during calibration, °K.

B_{wv} = Water vapor content of the bag sample, as analyzed, volume fraction.

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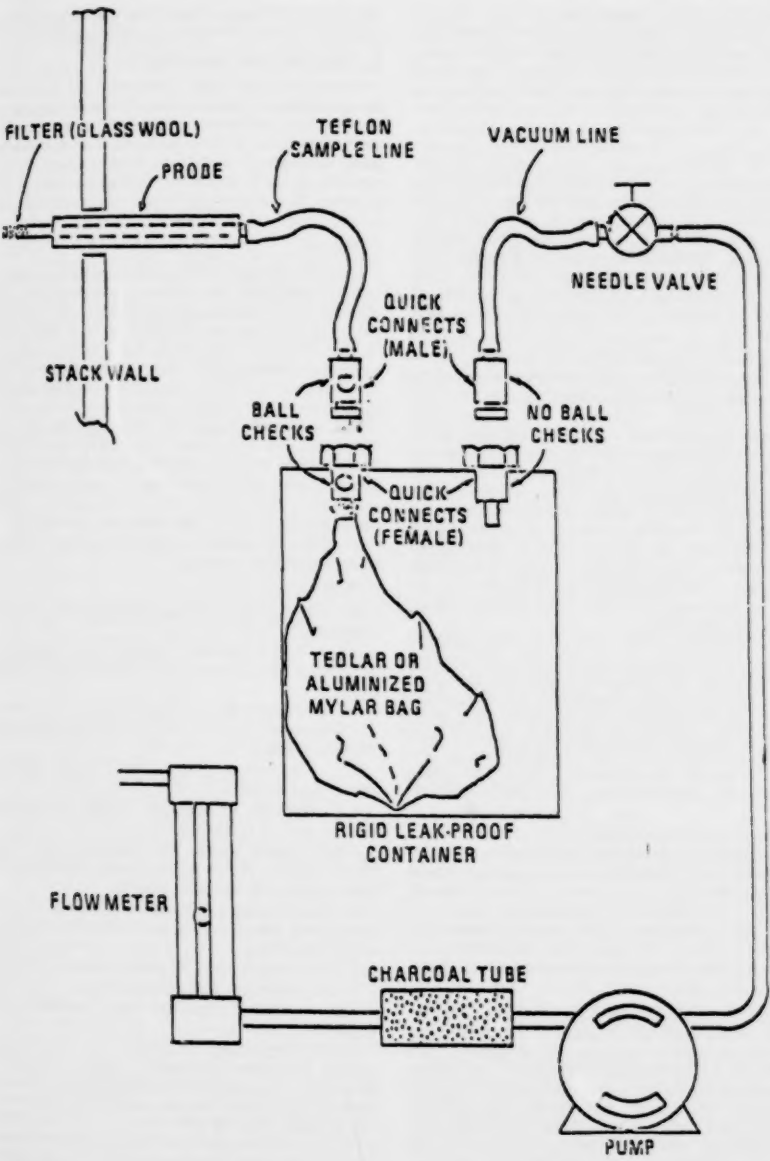


Figure 106-1. Integrated-bag sampling train. (Mention of trade names or specific products does not constitute endorsement by the Environmental Protection Agency.)

METHOD 107—DETERMINATION OF VINYL CHLORIDE CONTENT OF INPROCESS WASTEWATER SAMPLES, AND VINYL CHLORIDE CONTENT OF POLYVINYL CHLORIDE RESIN, SLURRY, WET CAKE, AND LATEX SAMPLES

Introduction

Performance of this method should not be attempted by persons unfamiliar with the operation of a gas chromatograph (GC), nor by those who are unfamiliar with source sampling, because knowledge beyond the scope of this presentation is required. Care must be exercised to prevent exposure of sampling personnel to vinyl chloride, a carcinogen.

1. Applicability and Principle

1.1 Applicability. This method applies to the measurement of the vinyl chloride monomer (VCM) content of inprocess wastewater samples, and the residual vinyl chloride monomer (RVCM) content of polyvinyl chloride (PVC) resins, wet cake, slurry, and latex samples. It cannot be used for polymer in fused forms, such as sheet or cubes. This method is not acceptable where methods from Section 304(h) of the Clean Water Act, 33 U.S.C. 1251 et seq. (the Federal Water Pollution Control Amendments of 1972 as amended by the Clean Water Act of 1977) are required.

1.2 Principle. The basis for this method relates to the vapor equilibrium that is established at a constant known temperature in a closed system between RVCM, PVC resin, water, and air. The RVCM in a PVC resin will equilibrate rapidly in a closed vessel, provided that the temperature of the PVC resin is maintained above the glass transition temperature of that specific resin.

2. Range and Sensitivity

The lower limit of detection of vinyl chloride will vary according to the sampling and chromatographic system. The system should be capable of producing a measurement for a 50-ppm vinyl chloride standard that is at least 10 times the standard deviation of the system background noise level.

3. Interferences

The chromatograph columns and the corresponding operating parameters herein described normally provide an adequate resolution of vinyl chloride; however, resolution interferences may be encountered on some sources. Therefore, the chromatograph operator shall select the column and operating parameters best suited to his particular analysis requirements, subject to the approval of the Administrator. Approval is automatic provided that the tester produces confirming data through an adequate supplemental analytical technique, such as analysis with a different column or GC/

mass spectroscopy, and has the data available for review by the Administrator.

4. Precision and Reproducibility

An interlaboratory comparison between seven laboratories of three resin samples, each split into three parts, yielded a standard deviation of 2.63 percent for a sample with a mean of 2.09 ppm, 4.16 percent for a sample with a mean of 1.66 ppm, and 5.29 percent for a sample with a mean of 62.66 ppm.

5. Safety

Do not release vinyl chloride to the laboratory atmosphere during preparation of standards. Venting or purging with VCM/air mixtures must be held to a minimum. When they are required, the vapor must be routed to outside air. Vinyl chloride, even at low ppm levels, must never be vented inside the laboratory. After vials have been analyzed, the gas must be vented prior to removal of the vial from the instrument turntable. Vials must be vented through a hypodermic needle connected to an activated charcoal tube to prevent release of vinyl chloride into the laboratory atmosphere. The charcoal must be replaced prior to vinyl chloride breakthrough.

6. Apparatus

6.1 Sampling. The following equipment is required:

6.1.1 Glass bottles. 60-ml (2-oz) capacity, with wax-lined screw-on tops, for PVC samples.

6.1.2 Glass Vials. Headspace vials, with Teflon-faced butyl rubber sealing discs, for water samples.

6.1.3 Adhesive Tape. To prevent loosening of bottle tops.

6.2 Sample Recovery. The following equipment is required:

6.2.1 Glass Vials. Headspace vials, with butyl rubber septa and aluminum caps. Silicone rubber is not acceptable.

6.2.2 Analytical Balance. Capable of determining sample weight within an accuracy of ± 1 percent.

6.2.3 Vial Sealer. To seal headspace vials.

6.2.4 Syringe. 100- μ l capacity.

6.3 Analysis. The following equipment is required:

6.3.1 Headspace Sampler and Chromatograph. Capable of sampling and analyzing a constant amount of headspace gas from a sealed vial, while maintaining that vial at a temperature of $90^{\circ}\text{C} \pm 0.5^{\circ}\text{C}$. The chromatograph shall be equipped with a flame ionization detector. Perkin-Elmer Corporation Models F-40, F-42, F-45, HS-6, and HS-100, and Hewlett-Packard Corporation Model 19395A have been found satisfactory. Chromatograph backflush capability may be required.

6.3.2 Chromatographic Columns. Stainless steel 1 m by 3.2 mm and 2 m by 3.2 mm, both containing 50/80-mesh Porapak Q. The analyst may use other columns provided that the precision and accuracy of the analysis of vinyl chloride standards are not impaired and he has available for review information confirming that there is adequate resolution of the vinyl chloride peak. (Adequate resolution is defined as an area overlap of not more than 10 percent of the vinyl chloride peak by an interferent peak. Calculation of area overlap is explained in Appendix C, Procedure 1: "Determination of Adequate Chromatographic Peak Resolution.") Two 1.83 m columns, each containing 1 percent Carbowax 1500 on Carbowax B, have been suggested for samples containing acetaldehyde.

6.3.3 Thermometer. 0 to 100°C, accurate to $\pm 0.1^\circ\text{C}$.

6.3.4 Integrator-Recorder. To record chromatograms.

6.3.5 Barometer. Accurate to ± 1 mm Hg.

6.3.6 Regulators. For required gas cylinders.

6.3.7 Headspace Vial Pre-Pressurizer. Nitrogen pressurized hypodermic needle inside protective shield. (Blueprint available from Test Support Section, Emission Measurement Branch, Office of Air Quality Planning and Standards, Environmental Protection Agency, Mail Drop 19, Research Triangle Park, N.C. 27711.)

7. Reagents

Use only reagents that are of chromatographic grade.

7.1 Analysis. The following items are required for analysis:

7.1.1 Hydrogen. Zero grade.

7.1.2 Nitrogen or Helium. Zero grade.

7.1.3 Air. Zero grade.

7.1.4 Water. Interference free.

7.2 Calibration. The following items are required for calibration:

7.2.1 Cylinder Standards (4) Gas mixture standards (50-, 500-, 2000- and 4000-ppm vinyl chloride in nitrogen cylinders). The tester may use cylinder standards to directly prepare a chromatograph calibration curve as described in Section 9.2, if the following conditions are met: (a) The manufacturer certifies the gas composition with an accuracy of ± 3 percent or better (see Section 7.2.1.1). (b) The manufacturer recommends a maximum shelf life over which the gas concentration does not change by greater than ± 5 percent from the certified value. (c) The manufacturer affixes the date of gas cylinder preparation, certified vinyl chloride concentration, and recommended maximum shelf life to the cylinder before shipment to the buyer.

7.2.1.1 Cylinder Standards Certification. The manufacturer shall certify the concen-

tration of vinyl chloride in nitrogen in each cylinder by (a) directly analyzing each cylinder and (b) calibrating his analytical procedure on the day of cylinder analysis. To calibrate his analytical procedure, the manufacturer shall use, as a minimum, a 3-point calibration curve. It is recommended that the manufacturer maintain (1) a high-concentration calibration standard (between 4000 and 8000 ppm) to prepare his calibration curve by an appropriate dilution technique and (2) a low-concentration calibration standard (between 50 and 500 ppm) to verify the dilution technique used. If the difference between the apparent concentration read from the calibration curve and the true concentration assigned to the low-concentration calibration standard exceeds 5 percent of the true concentration, the manufacturer shall determine the source of error and correct it, then repeat the 3-point calibration.

7.2.1.2 Verification of Manufacturer's Calibration Standards. Before using, the manufacturer shall verify each calibration standard by (a) comparing it to gas mixtures prepared (with 99 mole percent vinyl chloride) in accordance with the procedure described in Section 7.1 of Method 106 or by (b) calibrating it against vinyl chloride cylinder Standard Reference Materials (SRM's) prepared by the National Bureau of Standards, if such SRM's are available. The agreement between the initially determined concentration value and the verification concentration value must be within 5 percent. The manufacturer must reverify all calibration standards on a time interval consistent with the shelf life of the cylinder standards sold.

8. Procedure

8.1 Sampling.

8.1.1 PVC Sampling. Allow the resin or slurry to flow from a tap on the tank or silo until the tap line has been well purged. Extend and fill a 60-ml sample bottle under the tap, and immediately tighten a cap on the bottle. Wrap adhesive tape around the cap and bottle to prevent the cap from loosening. Place an identifying label on each bottle, and record the date, time, and sample location both on the bottles and in a log book. All samples should be kept refrigerated.

8.1.2 Water Sampling. At the sampling location fill the vials bubble-free to overflowing so that a convex meniscus forms at the top. The excess water is displaced as the sealing disc is carefully placed, with the Teflon side down, on the opening of the vial.

Place the aluminum seal over the disc and the neck of the vial, and crimp into place. Affix an identifying label on the bottle, and record the date, time, and sample location

both on the vials and in a log book. All samples must be kept refrigerated until analyzed.

8.2 Sample Recovery. Samples must be run within 24 hours.

8.2.1 Resin Samples. The weight of the resin used must be between 0.1 and 4.5 grams. An exact weight must be obtained (± 1 percent) for each sample. In the case of suspension resins, a volumetric cup can be prepared for holding the required amount of sample. When the cup is used, open the sample bottle, and add the cup volume of resin to the tared sample vial (tared, including septum and aluminum cap). Obtain the exact sample weight, add 100 μ l or about two equal drops of water, and immediately seal the vial. Report this value on the data sheet; it is required for calculation of RVC. In the case of dispersion resins, the cup cannot be used. Weigh the sample in an aluminum dish, transfer the sample to the tared vial, and accurately weigh it in the vial. After prepressurization of the samples, condition them for a minimum of 1 hour in the 90° C bath. Do not exceed 5 hours.

Prepressurization is not required if the sample weight, as analyzed, does not exceed 0.2 gram. It is also not required if solution of the prepressurization equation yields an absolute prepressurization value that is within 30 percent of the atmospheric pressure.

NOTE: Some aluminum vial caps have a center section that must be removed prior to placing into sample tray. If the cap is not removed, the injection needle will be damaged.

8.2.2 Suspension Resin Slurry and Wet Cake Samples. Decant the water from a wet cake sample, and turn the sample bottle upside down onto a paper towel. Wait for the water to drain, place approximately 0.2 to 4.0 grams of the wet cake sample in a tared vial (tared, including septum and aluminum cap) and seal immediately. Then determine the sample weight (± 1 percent). All samples, weighing over 0.2 gram, must be prepressurized prior to conditioning for 1 hour at 90° C, except as noted in Section 8.2.1. A sample of wet cake is used to determine total solids (TS). This is required for calculating the RVC.

8.2.3 Dispersion Resin Slurry and Geon Latex Samples. The materials should not be filtered. Sample must be thoroughly mixed. Using a tared vial (tared, including septum and aluminum cap) add approximately eight drops (0.25 to 0.35 g) of slurry or latex using a medicine dropper. This should be done immediately after mixing. Seal the vial as soon as possible. Determine sample weight (± 1 percent). Condition the vial for 1 hour at 90° C in the analyzer bath. Determine the TS on the slurry sample (Section 8.3.5).

8.2.4 Inprocess Wastewater Samples. Using a tared vial (tared, including septum and aluminum cap) quickly add approximately 1 cc of water using a medicine dropper. Seal the vial as soon as possible. Determine sample weight (± 1 percent). Condition the vial for 1 hour at 90° C in the analyzer bath.

8.3 Analysis.

8.3.1 Preparation of Equipment. Install the chromatographic column and condition overnight at 160° C. In the first operation, Porapak columns must be purged for 1 hour at 230° C.

Do not connect the exit end of the column to the detector while conditioning. Hydrogen and air to the detector must be turned off while the column is disconnected.

8.3.1.1 Flow Rate Adjustments. Adjust flow rates as follows:

a. Nitrogen Carrier Gas. Set regulator on cylinder to read 50 psig. Set regulator on chromatograph to produce a flow rate of 30.0 cc/min. Accurately measure the flow rate at the exit end of the column using the soap film flowmeter and a stopwatch, with the oven and column at the analysis temperature. After the instrument program advances to the "B" (backflush) mode, adjust the nitrogen pressure regulator to exactly balance the nitrogen flow rate at the detector as was obtained in the "A" mode.

b. Vial Prepressurizer Nitrogen. After the nitrogen carrier is set, solve the following equation and adjust the pressure on the vial prepressurizer accordingly.

$$P = \frac{T_1}{T_2} \left[P_1 - \frac{P_{w1} - P_{w2}}{7.50} \right] - 10 \text{ k Pa}$$

Where:

T_1 = Ambient temperature, °K.

T_2 = Conditioning bath temperature, °K.

P_1 = Gas chromatograph absolute dosing pressure (analysis mode), k Pa.

P_{w1} = Water vapor pressure @ 90° C (525.8 mm Hg).

P_{w2} = Water vapor pressure @ 22° C (19.8 mm Hg).

7.50 = mm Hg per k Pa.

10 k Pa = Factor to adjust the prepressurized pressure to slightly less than the dosing pressure.

Because of gauge errors, the apparatus may over-pressurize the vial. If the vial pressure is at or higher than the dosing pressure, an audible double injection will occur. If the vial pressure is too low, errors will occur on resin samples because of inad-

equate time for head-space gas equilibrium. This condition can be avoided by running several standard gas samples at various pressures around the calculated pressure, and then selecting the highest pressure that does not produce a double injection. All samples and standards must be pressurized for 60 seconds using the vial prepressurizer. The vial is then placed into the 90° C conditioning bath and tested for leakage by placing a drop of water on the septum at the needle hole. A clean, burr-free needle is mandatory.

c. **Burner Air Supply.** Set regulator on cylinder to read 50 psig. Set regulator on chromatograph to supply air to burner at a rate between 250 and 300 cc/min. Check with bubble flowmeter.

d. **Hydrogen Supply.** Set regulator on cylinder to read 30 psig. Set regulator on chromatograph to supply approximately 35 ± 5 cc/min. Optimize hydrogen flow to yield the most sensitive detector response without extinguishing the flame. Check flow with bubble meter and record this flow.

8.3.1.2 **Temperature Adjustments.** Set temperatures as follows:

- a. Oven (chromatograph column), 140° C.
- b. Dosing Line, 150° C.
- c. Injection Block, 170° C.
- d. Sample Chamber, Water Temperature, 90° C ± 1.0 ° C.

8.3.1.3 **Ignition of Flame Ionization Detector.** Ignite the detector according to the manufacturer's instructions.

8.3.1.4 **Amplifier Balance.** Balance the amplifier according to the manufacturer's instructions.

8.3.2 **Programming the Chromatograph.** Program the chromatograph as follows:

- a. **I—Dosing or Injection Time.** The normal setting is 2 seconds.
- b. **A—"Analysis Time."** The normal setting is approximately 70 percent of the VCM retention time. When this timer terminates, the programmer initiates backflushing of the first column.
- c. **B—Backflushing Time.** The normal setting is double the "analysis time."
- d. **W—Stabilization Time.** The normal setting is 0.5 min to 1.0 min.
- e. **X—Number of Analyses Per Sample.** The normal setting is one.

8.3.3 **Preparation of Sample Turntable.** Before placing any sample into turntable, be certain that the center section of the aluminum cap has been removed. The numbered sample vials should be placed in the corresponding numbered positions in the turntable. Insert samples in the following order:

Position 1 and 2—Old 2000-ppm standards for conditioning. These are necessary only after the analyzer has not been used for 24 hours or longer.

Position 3—50-ppm standard, freshly prepared.

Position 4—500-ppm standard, freshly prepared.

Position 5—2000-ppm standard, freshly prepared.

Position 6—4000-ppm standard, freshly prepared.

Position 7—Sample No. 7 (This is the first sample of the day, but is given as 7 to be consistent with the turntable and the integrator printout.)

After all samples have been positioned, insert the second set of 50-, 500-, 2000-, and 4000-ppm standards. Samples, including standards, must be conditioned in the bath of 90° C for 1 hour (not to exceed 5 hours).

8.3.4 **Start Chromatograph Program.** When all samples, including standards, have been conditioned at 90° C for 1 hour, start the analysis program according to the manufacturer's instructions. These instructions must be carefully followed when starting and stopping a program to prevent damage to the dosing assembly.

8.3.5 **Determination of TS.** For wet cake, slurry, resin solution, and PVC latex samples, determine TS for each sample by accurately weighing approximately 3 to 4 grams of sample in an aluminum pan before and after placing in a draft oven (105 to 110° C). Samples must be dried to constant weight. After first weighing, return the pan to the oven for a short period of time, and then reweigh to verify complete dryness. The TS are then calculated as the final sample weight divided by initial sample weight.

9. Calibration

Calibration is to be performed each 8-hour period the chromatograph is used. Alternatively, calibration with duplicate 504, 500-, 2,000-, and 4,000-ppm standards (hereafter described as a four-point calibration) may be performed on a monthly basis, provided that a calibration confirmation test consisting of duplicate analyses of an appropriate standard is performed once per plant shift, or once per chromatograph carousel operation (if the chromatograph operation is less frequent than once per shift). The criterion for acceptance of each calibration confirmation test is that both analyses of 500-ppm standards [2,000-ppm standards if dispersion resin (excluding latex resin) samples are being analyzed] must be within 5 percent of the most recent four-point calibration curve. If this criterion is not met, then a complete four-point calibration must be performed before sample analyses can proceed.

9.1 **Preparation of Standards.** Calibration standards are prepared as follows: Place 100 μ l or about two equal drops of distilled water in the sample vial, then fill the vial with the VCM/nitrogen standard, rapidly seat the septum, and seal with the aluminum cap. Use a $\frac{1}{8}$ -in. stainless steel line

from the cylinder to the vial. Do not use rubber or Tygon tubing. The sample line from the cylinder must be purged (into a properly vented hood) for several minutes prior to filling the vials. After purging, reduce the flow rate to 500 to 1000 cc/min. Place end of tubing into vial (near bottom). Position a septum on top of the vial, pressing it against the 1/8-in. filling tube to minimize the size of the vent opening. This is necessary to minimize mixing air with the standard in the vial. Each vial is to be purged with standard for 90 seconds, during which time the filling tube is gradually slid to the top of the vial. After the 90 seconds, the tube is removed with the septum, simultaneously sealing the vial. Practice will be necessary to develop good technique. Rubber gloves should be worn during the above operations. The sealed vial must then be pressurized for 60 seconds using the vial prepressurizer. Test the vial for leakage by placing a drop of water on the septum at the needle hole. Prepressurization of standards is not required unless samples have been prepressurized.

9.2 Preparation of Chromatograph Calibration Curve.

Prepare two vials each of 50-, 500-, 2,000-, and 4,000-ppm standards. Run the calibration samples in exactly the same manner as regular samples. Plot A_s , the integrator area counts for each standard sample, versus C_s , the concentration of vinyl chloride in each standard sample. Draw a straight line

through the points derived by the least squares method.

10. Calculations

10.1 Response Factor. If the calibration curve described in Section 9.2 passes through zero, an average response factor, R_s , may be used to facilitate computation of vinyl chloride sample concentrations.

To compute R_s , first compute a response factor, R_s , for each sample as follows:

$$R_s = \frac{A_s}{C_s} \quad \text{Eq. 107-1}$$

where:

R_s = Response factor, area counts/ppm.

A_s = Chromatogram area counts of vinyl chloride for the sample, area counts.

C_s = Concentration of vinyl chloride in the standard sample, ppm.

Sum the individual response factors, and calculate R_s . If the calibration curve does not pass through zero, use the calibration curve to determine each sample concentration.

10.2 Residual Vinyl Chloride Monomer Concentration, (C_{rv}) or Vinyl Chloride Monomer Concentration. Calculate C_{rv} in ppm or mg/kg as follows:

$$C_{rv} = \frac{A_s P_a}{R_f T_1} \left[\frac{M_v V_g}{R m} + K_p (TS) T_2 + K_w (1 - TS) T_2 \right] \quad \text{Eq. 107-2}$$

Where:

A_s = Chromatogram area counts of vinyl chloride for the sample.

P_a = Ambient atmospheric pressure, mm Hg.

R_f = Response factor in area counts per ppm VCM.

T_1 = Ambient laboratory temperature, °K.

M_v = Molecular weight of VCM, 62.5 g/mole.

V_g = Volume of vapor phase, cm^3

$$= V_v - \frac{m(TS)}{1.36} - \frac{m(1-TS)}{0.9653}$$

R = Gas constant, (62360 cm^3) (mm Hg)/(mole)(°K).

m = Sample weight, g.

K_p = Henry's Law Constant for VCM in PVC @ 90° C, $6.52 \times 10^{-6} \text{ g/g/mm Hg}$.

TS = Total solids expressed as a decimal fraction.

T_2 = Equilibrium temperature, °K.

K_s = Henry's Law Constant for VCM in water @ 90°C, 7×10^{-7} g/g/mm Hg.

V_v = Vial volume, cm^3 .

1.36 = Density of PVC at 90°C, g/cm^3 .

0.9653 = Density of water at 90°C, g/cm^3 .

$$= V_v - \frac{m(\text{TS})}{1.36} - \frac{m(1-\text{TS})}{0.9653}$$

Results calculated using these equations represent concentration based on the total sample. To obtain results based on dry PVC content, divide by TS.

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METHOD 107A—DETERMINATION OF VINYL CHLORIDE CONTENT OF SOLVENTS, RESIN, SOLVENT SOLUTION, POLYVINYL CHLORIDE RESIN, RESIN SLURRY, WET RESIN, AND LATEX SAMPLES

Introduction

Performance of this method should not be attempted by persons unfamiliar with the operation of a gas chromatograph (GC) or by those who are unfamiliar with source sampling because knowledge beyond the scope of this presentation is required. Care must be exercised to prevent exposure of sampling personnel to vinyl chloride, a carcinogen.

1. Applicability and Principle

1.1 Applicability. This is an alternative method and applies to the measurement of the vinyl chloride content of solvents, resin solvent solutions, polyvinyl chloride (PVC) resin, wet cake slurries, latex, and fabricated resin samples. This method is not acceptable where methods from Section 304(h) of

the Clean Water Act, 33 U.S.C. 1251 et seq., (the Federal Water Pollution Control Act Amendments of 1972 as amended by the Clean Water Act of 1977) are required.

1.2 Principle. The basis for this method lies in the direct injection of a liquid sample into a chromatograph and the subsequent evaporation of all volatile material into the carrier gas stream of the chromatograph, thus permitting analysis of all volatile material including vinyl chloride.

2. Range and Sensitivity

The lower limit of detection of vinyl chloride in dry PVC resin is 0.2 ppm. For resin solutions, latexes, and wet resin, this limit rises inversely as the nonvolatile (resin) content decreases.

With proper calibration, the upper limit may be extended as needed.

3. Interferences

The chromatograph columns and the corresponding operating parameters herein described normally provide an adequate resolution of vinyl chloride. In cases where resolution interferences are encountered, the chromatograph operator shall select the column and operating parameters best suited to his particular analysis problem, subject to the approval of the Administrator. Approval is automatic, provided that the tester produces confirming data through an adequate supplemental analytical technique, such as analysis with a different column or GC/mass spectroscopy, and has the data available for review by the Administrator.

4. Precision and Reproducibility

A standard sample of latex containing 181.8 ppm vinyl chloride analyzed 10 times by the alternative method showed a standard deviation of 7.5 percent and a mean error of 0.21 percent.

A sample of vinyl chloride copolymer resin solution was analyzed 10 times by the alternative method and showed a standard deviation of 6.6 percent at a level of 35 ppm.

5. Safety

Do not release vinyl chloride to the laboratory atmosphere during preparation of standards. Venting or purging with vinyl chloride monomer (VCM) air mixtures must be held to minimum. When purging is required, the vapor must be routed to outside

air. Vinyl chloride, even at low-ppm levels, must never be vented inside the laboratory.

6. Apparatus

6.1 Sampling. The following equipment is required:

6.1.1 Glass Bottles. 16-oz wide mouth wide polyethylene-lined, screw-on tops.

6.1.2 Adhesive Tape. To prevent loosening of bottle tops.

6.2 Sample Recovery. The following equipment is required:

6.2.1 Glass Vials. 20-ml capacity with polycone screw caps.

6.2.2 Analytical Balance. Capable of weighing to ± 0.01 gram.

6.2.3 Syringe. 50-microliter size, with removable needle.

6.2.4 Fritted Glass Sparger. Fine porosity.

6.2.5 Aluminum Weighing Dishes.

6.2.6 Sample Roller or Shaker. To help dissolve sample.

6.3 Analysis. The following equipment is required:

6.3.1 Gas Chromatograph. Hewlett Packard Model 5720A or equivalent.

6.3.2 Chromatograph Column. Stainless steel, 6.1 m by 3.2 mm, packed with 20 percent Tergitol E-35 on Chromosorb W AW 60/80 mesh. The analyst may use other columns provided that the precision and accuracy of the analysis of vinyl chloride standards are not impaired and that he has available for review information confirming that there is adequate resolution of the vinyl chloride peak. (Adequate resolution is defined as an area overlap of not more than 10 percent of the vinyl chloride peak by an interfering peak. Calculation of area overlap is explained in Appendix C, Procedure 1: "De-

termination of Adequate Chromatographic Peak Resolution.")

6.3.3 Valco Instrument Six-Port Rotary Valve. For column back flush.

6.3.4 Septa. For chromatograph injection port.

6.3.5 Injection Port Liners. For chromatograph used.

6.3.6 Regulators. For required gas cylinders.

6.3.7 Soap Film Flowmeter. Hewlett Packard No. 0101-0113 or equivalent.

6.4 Calibration. The following equipment is required:

6.4.1 Analytical Balance. Capable of weighing to ± 0.0001 g.

6.4.2 Erlenmeyer Flask With Glass Stopper. 125 ml.

6.4.3 Pipets. 0.1, 0.5, 1, 5, 10, and 50 ml.

6.4.4 Volumetric Flasks. 10 and 100 ml.

7. Reagents

Use only reagents that are of chromatograph grade.

7.1 Analysis. The following items are required:

7.1.1 Hydrogen Gas. Zero grade.

7.1.2 Nitrogen Gas. Zero grade.

7.1.3 Air. Zero grade.

7.1.4 Tetrahydrofuran (THF). Reagent grade.

Analyze the THF by injecting 10 microliters into the prepared gas chromatograph. Compare the THF chromatogram with that shown in Figure 107A-1. If the chromatogram is comparable to A, the THF should be sparged with pure nitrogen for approximately 2 hours using the fritted glass sparger to attempt to remove the interfering peak. Reanalyze the sparged THF to determine whether the THF is acceptable for use. If the scan is comparable to B, the THF should be acceptable for use in the analysis.

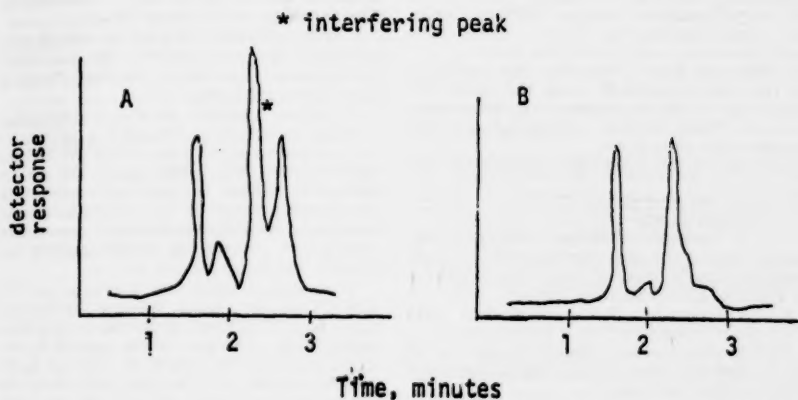


Figure 107A-1

7.1.5 N, N-Dimethylacetamide (DMAC). Spectrographic grade. For use in place of THF.

7.2 Calibration. The following item is required:

7.2.1 Vinyl Chloride 99.9 Percent. Ideal Gas Products lecture bottle, or equivalent. For preparation of standard solutions.

8. Procedure

8.1 Sampling. Allow the liquid or dried resin to flow from a tap on the tank, silo, or pipeline until the tap has been purged. Fill a wide-mouth pint bottle, and immediately tightly cap the bottle. Place an identifying label on each bottle and record the date, time, sample location, and material.

8.2 Sample Treatment. Sample must be run within 24 hours.

8.2.1 Resin Samples. Weigh 9.00 ± 0.01 g of THF or DMAC in a tared 20-ml vial. Add 1.00 ± 0.01 g of resin to the tared vial containing the THF or DMAC. Close the vial tightly with the screw cap, and shake or otherwise agitate the vial until complete solution of the resin is obtained. Shaking may require several minutes to several hours, depending on the nature of the resin.

8.2.2 Suspension Resin Slurry and Wet Resin Sample. Slurry must be filtered using a small Buchner funnel with vacuum to yield a wet resin sample. The filtering process must be continued only as long as a steady stream of water is exiting from the funnel. Excessive filtration time could result in some loss of VCM. The wet resin sample is weighed into a tared 20-ml vial with THF or DMAC as described earlier for resin sam-

ples (8.2.1) and treated the same as the resin sample. A sample of the wet resin is used to determine total solids as required for calculating the residual VCM (Section 8.3.4).

8.2.3 Latex and Resin Solvent Solutions. Samples must be thoroughly mixed. Weigh 1.00 ± 0.01 g of the latex or resin-solvent solution into a 20-ml vial containing 9.00 ± 0.01 g of THF or DMAC as for the resin samples (8.2.1). Cap and shake until complete solution is obtained. Determine the total solids of the latex or resin solution sample (Section 8.3.4).

8.2.4 Solvents and Non-viscous Liquid Samples. No preparation of these samples is required. The neat samples are injected directly into the GC.

8.3 Analysis.

8.3.1 Preparation of GC. Install the chromatographic column, and condition overnight at 70°C . Do not connect the exit end of the column to the detector while conditioning.

8.3.1.1 Flow Rate Adjustments. Adjust the flow rate as follows:

a. Nitrogen Carrier Gas. Set regulator on cylinder to read 60 psig. Set column flow controller on the chromatograph using the soap film flowmeter to yield a flow rate of 40 cc/min.

b. Burner Air Supply. Set regulator on the cylinder at 40 psig. Set regulator on the chromatograph to supply air to the burner to yield a flow rate of 250 to 300 cc/min using the flowmeter.

c. Hydrogen. Set regulator on cylinder to read 60 psig. Set regulator on the chromato-

graph to supply 30 to 40 cc/min using the flowmeter. Optimize hydrogen flow to yield the most sensitive detector response without extinguishing the flame. Check flow with flowmeter and record this flow.

d. Nitrogen Back Flush Gas. Set regulator on the chromatograph using the soap film flowmeter to yield a flow rate of 40 cc/min.

8.3.1.2 Temperature Adjustments. Set temperature as follows:

a. Oven (chromatographic column) at 70° C.

b. Injection Port at 100° C.

c. Detector at 300° C.

8.3.1.3 Ignition of Flame Ionization Detector. Ignite the detector according to the manufacturer's instructions. Allow system to stabilize approximately 1 hour.

8.3.1.4 Recorder. Set pen at zero and start chart drive.

8.3.1.5 Attenuation. Set attenuation to yield desired peak height depending on sample VCM content.

8.3.2 Chromatographic Analyses.

a. Sample Injection. Remove needle from 50-microliter syringe. Open sample vial and draw 50-microliters of THF or DMAC sample recovery solution into the syringe. Recap sample vial. Attach needle to the syringe and while holding the syringe vertically (needle uppermost), eject 40 microliters into an absorbent tissue. Wipe needle with tissue. Now inject 10 microliters into chromatograph system. Repeat the injection until two consecutive values for the height of the vinyl chloride peak do not vary more than 5 percent. Use the average value for these two peak heights to compute the sample concentration.

b. Back Flush. After 4 minutes has elapsed after sample injection, actuate the back flush valve to purge the first 4 feet of the chromatographic column of solvent and other high boilers.

c. Sample Data. Record on the chromatograph strip chart the data from the sample label.

d. Elution Time. Vinyl chloride elutes at 2.8 minutes. Acetaldehyde elutes at 3.7 minutes. Analysis is considered complete when chart pen becomes stable. After 5 minutes, reset back flush valve and inject next sample.

8.3.3 Chromatograph Servicing.

a. Septum. Replace after five sample injections.

b. Sample Port Liner. Replace the sample port liner with a clean spare after five sample injections.

c. Chromatograph Shutdown. If the chromatograph has been shut down overnight, rerun one or more samples from the preceding day to test stability and precision prior to starting on the current day's work.

8.3.4 Determination of Total Solids (TS). For wet resin, resin solution, and PVC latex samples, determine the TS for each sample

by accurately weighing approximately 3 to 5 grams of sample into a tared aluminum pan. The initial procedure is as follows:

a. Where water is the major volatile component: Tare the weighing dish, and add 3 to 5 grams of sample to the dish. Weigh to the nearest milligram.

b. Where volatile solvent is the major volatile component: Transfer a portion of the sample to a 20-ml screw cap vial and cap immediately. Weigh the vial to the nearest milligram. Uncap the vial and transfer a 3- to 5-gram portion of the sample to a tared aluminum weighing dish. Recap the vial and reweigh to the nearest milligram. The vial weight loss is the sample weight.

To continue, place the weighing pan in a 130° C oven for 1 hour. Remove the dish and allow to cool to room temperature in a desiccator. Weigh the pan to the nearest 0.1 mg. Total solids is the weight of material in the aluminum pan after heating divided by the net weight of sample added to the pan originally times 100.

9. Calibration of the Chromatograph

9.1 Preparation of Standards. Prepare a 1 percent by weight (approximate) solution of vinyl chloride in THF or DMAC by bubbling vinyl chloride gas from a cylinder into a tared 125-ml glass-stoppered flask containing THF or DMAC. The weight of vinyl chloride to be added should be calculated prior to this operation, i.e., 1 percent of the weight of THF or DMAC contained in the tared flask. This must be carried out in a laboratory hood. Adjust the vinyl chloride flow from the cylinder so that the vinyl chloride dissolves essentially completely in the THF or DMAC and is not blown to the atmosphere. Take particular care not to volatilize any of the solution. Stopper the flask and swirl the solution to effect complete mixing. Weigh the stoppered flask to nearest 0.1 mg to determine the exact amount of vinyl chloride added.

Pipet 10 ml of the approximately 1 percent solution into a 100-ml glass-stoppered volumetric flask, and add THF or DMAC to fill to the mark. Cap the flask and invert 10 to 20 times. This solution contains approximately 1,000 ppm by weight of vinyl chloride (note the exact concentration).

Pipet 50-, 10-, 5-, 1-, 0.5-, and 0.1-ml aliquots of the approximately 1,000 ppm solution into 10 ml glass stoppered volumetric flasks. Dilute to the mark with THF or DMAC, cap the flasks and invert each 10 to 20 times. These solutions contain approximately 500, 100, 50, 10, 5, and 1 ppm vinyl chloride. Note the exact concentration of each one. These standards are to be kept under refrigeration in stoppered bottles, and must be renewed every 3 months.

9.2 Preparation of Chromatograph Calibration Curve.

Obtain the GC for each of the six final solutions prepared in Section 9.1 by using the procedure in Section 8.3.2. Prepare a chart plotting peak height obtained from the chromatogram of each solution versus the known concentration. Draw a straight line through the points derived by the least squares method.

10. Calculations

10.1 Response Factor. From the calibration curve described in Section 9.2, select the value of C_s that corresponds to H_s for each sample. Compute the response factor, R_s , for each sample as follows:

$$R_s = \frac{C_s}{H_s} \quad \text{Eq. 107A-1}$$

where:

R_s =Chromatograph response factor, ppm/mm.

C_s =Concentration of vinyl chloride in the standard sample, ppm.

H_s =Peak height of the standard sample, mm.

10.2 Residual vinyl chloride monomer concentration (C_{mv}) or vinyl chloride monomer concentration in resin:

$$C_{mv} = 10H_s R_s \quad \text{Eq. 107A-2}$$

Where:

C_{mv} =Concentration of residual vinyl chloride monomer, ppm.

H_s =Peak height of sample, mm.

R_s =Chromatograph response factor.

10.3 Samples containing volatile material, i.e., resin solutions, wet resin, and latexes:

$$C_{mv} = \frac{H_s R_s (1,000)}{TS} \quad \text{Eq. 107A-3}$$

where:

TS =Total solids in the sample, weight fraction.

10.4 Samples of solvents and in process wastewater:

$$C_{mv} = \frac{H_s R_s}{0.888} \quad \text{Eq. 107A-4}$$

Where:

0.888=Specific gravity of THF.

11. Bibliography

1. Communication from R. N. Wheeler, Jr.; Union Carbide Corporation. Part 61 National Emissions Standards for Hazardous Air Pollutants Appendix B, Method 107—Alternate Method, September 19, 1977.

METHOD 108—DETERMINATION OF PARTICULATE AND GASEOUS ARSENIC EMISSIONS

1. Applicability and Principle

1.1 Applicability. This method applies to the determination of inorganic arsenic (As) emissions from stationary sources as specified in the applicable subpart.

1.2 Principle. Particulate and gaseous arsenic emissions are withdrawn isokinetically from the source and collected on a glass mat filter and in water. The collected arsenic is then analyzed by means of atomic absorption spectrophotometry.

2. Apparatus

2.1 Sampling Train. A schematic of the sampling train is shown in Figure 108-1; it is similar to the Method 5 train of 40 CFR Part 60, Appendix A. Note: This and all subsequent references to other methods refer to the methods in 40 CFR Part 60, Appendix A. The sampling train consists of the following components:

2.1.1 Probe Nozzle, Probe Liner, Pitot Tube, Differential Pressure Gauge, Filter Holder, Filter Heating System, Metering System, Barometer, and Gas Density Determination Equipment. Same as Method 5, Sections 2.1.1 to 2.1.6 and 2.1.8 to 2.1.10, respectively.

2.1.2 Filter Heating System. Any heating (or cooling) system capable of maintaining a sample gas temperature at the exit end of the filter holder during sampling at $121 \pm 14^\circ\text{C}$ ($250 \pm 25^\circ\text{F}$). Install a temperature gauge capable of measuring temperature to within 3°C (5.4°F) at the exit end of the filter holder so that the sample gas temperature can be regulated and monitored during sampling. The tester may use systems other than the one shown in APTD-0591.

2.1.3 Impingers. Four impingers connected in series with leak-free ground-glass fittings or any similar leak-free noncontaminating fittings. For the first, third, and fourth impingers, use the Greenburg-Smith design, modified by replacing the tip with a 1.3-cm-ID (0.5 in.) glass tube extending to about 1.3 cm (0.5 in.) from the bottom of

the flask. For the second impinger, use the Greenburg-Smith design with the standard tip. The tester may use modifications (e.g., flexible connections between the impingers, materials other than glass, or flexible vacuum lines to connect the filter holder to

the condenser), subject to the approval of the Administrator.

Place a thermometer, capable of measuring temperature to within 1°C (2°F), at the outlet of the fourth impinger for monitoring purposes.

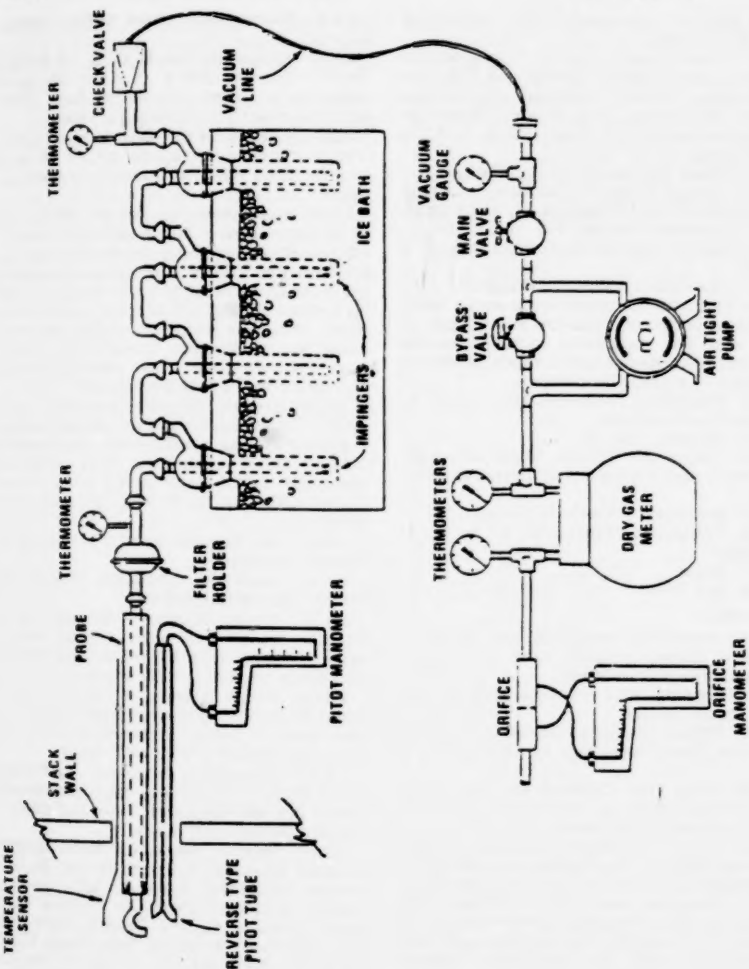


Figure 108-1. Arsenic sampling train.

2.2 Sample Recovery. The following items are needed:

2.2.1 Probe-Liner and Probe-Nozzle Brushes, Petri Dishes, Graduated Cylinder or Balance, Plastic Storage Containers, Rubber Policeman, and Funnel. Same as Method 5, Sections 2.2.1 and 2.2.4 to 2.2.8, respectively.

2.2.2 Wash Bottles. Polyethylene (2).

2.2.3 Sample Storage Containers. Chemically resistant, polyethylene or polypropylene for glassware washes, 500- or 1000-ml.

2.3 Analysis. The following equipment is needed:

2.3.1 Spectrophotometer. Equipped with an electrodeless discharge lamp and a background corrector to measure absorbance at 193.7 nm. For measuring samples having less than 10 $\mu\text{g As/ml}$, use a vapor generator accessory or a graphite furnace.

2.3.2 Recorder. To match the output of the spectrophotometer.

2.3.3 Beakers. 150-ml.

2.3.4 Volumetric Flasks. Glass 50-, 100-, 200-, 500-, and 1000-ml; and polypropylene, 50-ml.

2.3.5 Balance. To measure within 0.5 g.

2.3.6 Volumetric Pipets. 1-, 2-, 3-, 5-, 8-, and 10-ml.

2.3.7 Oven.

2.3.8 Hot Plate.

3. Reagents

Unless otherwise specified, use American Chemical Society reagent grade (or equivalent) chemicals throughout.

3.1 Sampling. The reagents used in sampling are as follows:

3.1.1 Filters. Same as Method 5 except that the filters need not be unreactive to SO_2 .

3.1.2 Silica Gel, Crushed Ice, and Stop-cock Grease. Same as Method 5, Sections 3.1.2, 3.1.4, and 3.1.5, respectively.

3.1.3 Water. Deionized distilled to meet American Society for Testing and Materials Specification D 1133-74, Type 3 (incorporated by reference—see § 60.17). When high concentrations of organic matter are not expected to be present, the analyst may omit the KMnO_4 test for oxidizable organic matter.

3.2 Sample Recovery. 0.1 N sodium hydroxide (NaOH) is required. Dissolve 4.00 g of NaOH in about 500 ml of water in a 1-liter volumetric flask. Then, dilute to exactly 1.0 liter with water.

3.3 Analysis. The reagents needed for analysis are as follows:

3.3.1 Water. Same as 3.1.3.

3.3.2 Sodium Hydroxide, 0.1 N. Same as 3.2.

3.3.3 Sodium Borohydride (NaBH_4), 5 Percent (W/V). Dissolve 5.00 g of NaBH_4 in about 500 ml of 0.1 N NaOH in a 1-liter volumetric flask. Then, dilute to exactly 1.0 liter with 0.1 N NaOH .

3.3.4 Hydrochloric Acid (HCl), Concentrated.

3.3.5 Potassium Iodide (KI), 30 Percent (W/V). Dissolve 300 g of KI in 500 ml of water in a 1-liter volumetric flask. Then, dilute to exactly 1.0 liter with water.

3.3.6 Nitric Acid (HNO_3), Concentrated.

3.3.7 Nitric Acid, 0.8 N. Dilute 52 ml of concentrated HNO_3 to exactly 1.0 liter with water.

3.3.8 Nitric Acid, 50 Percent (V/V). Add 50 ml concentrated HNO_3 to 50 ml water.

3.3.9 Stock Arsenic Standard, 1 mg As/ml. Dissolve 1.3203 g of primary standard grade As_2O_3 in 20 ml of 0.1 N NaOH in a 150-ml beaker. Slowly add 30 ml of concentrated HNO_3 . Heat the resulting solution and evaporate just to dryness. Transfer the residue quantitatively to a 1-liter volumetric flask and dilute to 1.0 liter with water.

3.3.10 Arsenic Working Solution, 1.0 $\mu\text{g As/ml}$. Pipet exactly 1.0 ml of stock arsenic standard into an acid-cleaned, appropriately labeled 1-liter volumetric flask containing about 500 ml of water and 5 ml of concentrated HNO_3 . Dilute to exactly 1.0 liter with water.

3.3.11 Air. Suitable quality for atomic absorption analysis.

3.3.12 Acetylene. Suitable quality for atomic absorption analysis.

3.3.13 Nickel Nitrate, 5 Percent (W/V). Dissolve 24.780 g of nickel nitrate hexahydrate in water in a 100-ml volumetric flask and dilute to 100 ml with water.

3.3.14 Nickel Nitrate, 1 Percent (W/V). Pipet 20 ml of 5 percent nickel nitrate solution into a 100-ml volumetric flask and dilute to exactly 100 ml with water.

3.3.15 Hydrogen Peroxide, 3 Percent. Pipet 50 ml of 30 percent hydrogen peroxide into a 500 ml volumetric flask and dilute to exactly 500 ml with water.

3.3.16 Quality Assurance Audit Samples. Arsenic samples prepared by the Environmental Protection Agency's (EPA) Environmental Systems Laboratory, Quality Assurance Division, Source Branch, Mail Drop 77A, Research Triangle Park, North Carolina 27711. Each set will consist of two vials of unknown concentrations. Only when making compliance determinations, obtain an audit sample set from the Quality Assurance Management Office at each EPA regional office or the responsible enforcement office. (Note: The tester should notify the Quality Assurance Office or the responsible enforcement agency at least 30 days prior to the test date to allow sufficient time for delivery.)

4. Procedure

4.1 Sampling. Because of the complexity of this method, testers must be trained and experienced with the test procedures in order to obtain reliable results.

4.1.1 Pretest Preparation. Follow the general procedure given in Method 5, Section 4.1.1, except the filter need not be weighed.

4.1.2 Preliminary Determinations. Follow the general procedure given in Method 5, Section 4.1.2, except select the nozzle size to maintain isokinetic sampling rates below 28 liters/inin (1.0 cfm).

4.1.3 Preparation of Collection Train. Follow the general procedure given in Method 5, Section 4.1.3.

4.1.4 Leak-Check Procedures. Follow the leak-check procedures given in Method 5,

Sections 4.1.4.1 (Pretest Leak-Check), 4.1.4.2 (Leak-Checks During Sample Run), and 4.1.4.3 (Post-Test Leak-Check).

4.1.5 Arsenic Train Operation. Follow the general procedure given in Method 5, Section 4.1.5, except maintain a temperature of 107° to 135°C (225° to 275°F) around the filter and maintain isokinetic sampling flow rates below 28 liters/min (1.0 cfm). For each run, record the data required on a data sheet such as the one shown in Figure 108-2m.

4.1.6 Calculation of Percent Isokinetic. Same as Method 5, Section 4.1.6.

PLANT _____
LOCATION _____
OPERATOR _____
DATE _____
RUN NO. _____
SAMPLE BOX NO. _____
METER BOX NO. _____
METER ΔH_g _____
C FACTOR _____
PILOT TUBE COEFFICIENT, C_p _____

SCHEMATIC OF STACK CROSS SECTION

AMBIENT TEMPERATURE _____
 BAROMETRIC PRESSURE _____
 ASSUMED MOISTURE, % _____
 PROBE LENGTH, m (ft) _____
 NOZZLE IDENTIFICATION NO. _____
 AVERAGE CALIBRATED NOZZLE DIAMETER, cm (in.) _____
 PROBE HEATER SETTING _____
 LEAK RATE, m³/min. (cfm) _____
 PROBE LINER MATERIAL _____

[illegible]

Figure 10B 2 Arsenic field data.

4.2 Sample Recovery. The same as Method 5, Section 4.2 except that 0.1 N NaOH is used as the cleanup solvent instead of acetone and that the impinger water is treated as follows:

Container Number 4 (Impinger Water). Clean each of the first two impingers and connecting glassware in the following manner:

a. Wipe the impinger ball joints free of silicone grease, and cap the joints.

b. Weigh the impinger and liquid to within ± 0.5 g. Record in the log the weight of liquid along with a notation of any color or film observed in the impinger catch. The weight of liquid is needed along with the silica gel data to calculate the stack gas moisture content.

c. Rotate and agitate each impinger, using the impinger contents as a rinse solution.

d. Transfer the liquid to Container Number 4. Remove the outlet ball-joint cap, and drain the contents through this opening. Do not separate the impinger parts (inner and outer tubes) while transferring their contents to the cylinder.

e. (Note: In Steps e and f below, measure and record the total amount of 0.1 N NaOH used for rinsing.) Pour approximately 30 ml of 0.1 N NaOH into each of the first two impingers, and agitate the impingers. Drain the 0.1 N NaOH through the outlet arm of each impinger into Container Number 4. Repeat this operation a second time; inspect the impingers for any abnormal conditions.

f. Wipe the ball joints of the glassware connecting the impingers and the back half of the filter holder free of silicone grease, and rinse each piece of glassware twice with 0.1 N NaOH; transfer this rinse into Container Number 4. (DO NOT RINSE or brush the glass-fritted filter support.) Mark the height of the fluid level to determine whether leakage occurs during transport. Label the container to identify clearly its contents.

4.2.1 Blanks. Save a portion of the 0.1 N NaOH used for cleanup as a blank. Take 200 ml of this solution directly from the wash bottle being used and place it in a plastic sample container labeled "NaOH blank." Also save a sample of the water, and place it in a container labeled "H₂O blank."

4.3 Arsenic Sample Preparation.

4.3.1 Container Number 1 (Filter). Place the filter and loose particulate matter in a 150-ml beaker. Also, add the filtered material from Container Number 2 (see Section 4.3.3). Add 50 ml of 0.1 N NaOH. Then stir and warm on a hot plate at low heat (do not boil) for about 15 minutes. Add 10 ml of concentrated HNO₃, bring to a boil, then simmer for about 15 minutes. Filter the solution through a glass fiber filter. Wash with hot water, and catch the filtrate in a clean 150-ml beaker. Boil the filtrate, and evaporate to dryness. Cool, add 5 ml of 50

percent HNO₃, and then warm and stir. Allow to cool. Transfer to a 50-ml volumetric flask, dilute to volume with water, and mix well.

4.3.2 Container Number 4 (Arsenic Impinger Sample).

Note: Prior to analysis, check the liquid level in Containers Number 2 and Number 4; confirm as to whether leakage occurred during transport on the analysis sheet. If a noticeable amount of leakage occurred, either void the sample or take steps, subject to the approval of the Administrator, to adjust the final results.

Transfer the contents of Container Number 4 to a 500-ml volumetric flask, and dilute to exactly 500 ml with water. Pipet 50 ml of the solution into a 150-ml beaker. Add 10 ml of concentrated HNO₃, bring to a boil, and evaporate to dryness. Allow to cool, add 5 ml of 50 percent HNO₃, and then warm and stir. Allow the solution to cool, transfer to a 50-ml volumetric flask, dilute to volume with water, and mix well.

4.3.3 Container Number 2 (Probe Wash). See note in 4.3.2 above. Filter (using a glass fiber filter) the contents of Container Number 2 into a 200-ml volumetric flask. Combine the filtered material with the contents of Container Number 1 (Filter).

Dilute the filtrate to exactly 200 ml with water. Then pipet 50 ml into a 150-ml beaker. Add 10 ml of concentrated HNO₃, bring to a boil, and evaporate to dryness. Allow to cool, add 5 ml of 50 percent HNO₃, and then warm and stir. Allow the solution to cool, transfer to a 50-ml volumetric flask, dilute to volume with water, and mix well.

4.3.4 Filter Blank. Determine a filter blank using two filters from each lot of filters used in the sampling. Cut each filter into strips, and treat each filter individually as directed in Section 4.3.1, beginning with the sentence, "Add 50 ml of 0.1 N NaOH."

4.3.5 0.1 N NaOH and Water Blanks. Treat separately 50 ml of 0.1 N NaOH and 50 ml water, as directed under Section 4.3.2, beginning with the sentence, "Pipet 50 ml of the solution into a 150-ml beaker."

4.4 Spectrophotometer Preparation. Turn on the power; set the wavelength, slit width, and lamp current; and adjust the background corrector as instructed by the manufacturer's manual for the particular atomic absorption spectrophotometer. Adjust the burner and flame characteristics as necessary.

4.5 Analysis.

4.5.1 Arsenic Determination. Prepare standard solutions as directed under Section 5.1, and measure their absorbances against 0.8 N HNO₃. Then, determine the absorbances of the filter blank and each sample using 0.8 N HNO₃ as a reference. If the sample concentration falls outside the range of the calibration curve, make an appropri-

ate dilution with 0.8 N HNO_3 so that the final concentration falls within the range of the curve. Determine the arsenic concentration in the filter blank (i.e., the average of the two blank values from each lot). Next, using the appropriate standard curve, determine the arsenic concentration in each sample fraction.

4.5.1.1 Arsenic Determination at Low Concentration. The lower limit of flame atomic absorption spectrophotometry is 10 $\mu\text{g As/ml}$. If the arsenic concentration of any sample is at a lower level, use the graphite furnace or vapor generator which is available as an accessory component. The analyst also has the option of using either of these accessories for samples whose concentrations are between 10 and 30 $\mu\text{g/ml}$. Follow the manufacturer's instructions in the use of such equipment.

4.5.1.1.1 Vapor Generator Procedure. Place a sample containing between 0 and 5 μg of arsenic in the reaction tube, and dilute to 15 ml with water. Since there is some trial and error involved in this procedure, it may be necessary to screen the samples by conventional atomic absorption until an approximate concentration is determined. After determining the approximate concentration, adjust the volume of the sample accordingly. Pipet 15 ml of concentrated HCl into each tube. Add 1 ml of 30 percent KI solution. Place the reaction tube into a 50°C water bath for 5 minutes. Cool to room temperature. Connect the reaction tube to the vapor generator assembly. When the instrument response has returned to baseline, inject 5.0 ml of 5 percent NaBH_4 , and integrate the resulting spectrophotometer signal over a 30-second time period.

4.5.1.1.2 Graphite Furnace Procedure. Dilute the digested sample so that a 5-ml aliquot contains less than 1.5 μg of arsenic. Pipet 5 ml of this digested solution into a 10-ml volumetric flask. Add 1 ml of the 1 percent nickel nitrate solution, 0.5 ml of 50 percent HNO_3 , and 1 ml of the 3 percent hydrogen peroxide and dilute to 10 ml with water. The sample is now ready to inject in the furnace for analysis.

Because instruments from different manufacturers are different, no detailed operating instructions will be given here. Instead, the analyst should follow the instructions provided with his particular instrument.

4.5.1.2 Check for Matrix Effects on the Arsenic Results. Same as Method 12, Section 5.4.2.

4.5.2 Container Number 3 (Silica Gel). The tester may conduct this step in the field. Weigh the spent silica gel (or silica gel plus impinger) to the nearest 0.5 g; record this weight.

4.6 Audit Analysis. Concurrently, analyze the two unknown audit samples with each set of compliance samples to evaluate the techniques of the analyst and the standards

preparation. (Note: It is recommended that known quality control samples be analyzed prior to the compliance and audit sample analysis to optimize the system's accuracy and precision. One source of these samples is the Source Branch listed in Section 3.3.16.) The same analyst, analytical reagents, and analytical system shall be used both for each set or sets of compliance samples and the EPA audit samples; if this condition is met, audit samples need not be included with any additional compliance analyses performed within the succeeding 30-day period for the same enforcement agency. An audit sample set may not be used to validate different sets of compliance samples under the jurisdiction of different enforcement agencies unless prior arrangements are made with both enforcement agencies.

Calculate the concentration in g/m^3 using the specified sample volume in the audit instructions. (Note: The analyst may determine immediately whether the audit analyses acceptable by reporting the audit results in g/m^3 and compliance results in $\mu\text{g/ml}$ by telephone.) Include the results of both audit samples, their identification numbers, and the analysts' names with the results of the compliance determination samples in appropriate reports to the EPA regional office or the appropriate enforcement agency. Include this information with subsequent compliance analyses for the same enforcement agency during the succeeding 30-day period.

5. Calibration

Maintain a laboratory log of all calibrations.

5.1 Standard Solutions. For the high level procedure pipet 1, 3, 5, 8, and 10 ml of the 1.0-mg As/ml stock solution into separate 100-ml volumetric flasks, each containing 5 ml of concentrated HNO_3 . If the low level vapor generator procedure is used, pipet 1, 2, 3, and 5 ml of 1.0 $\mu\text{g As/ml}$ standard solution into the separate reaction tubes. For the low level graphite furnace procedure, pipet 1, 5, 10 and 15 ml of 1.0 $\mu\text{g As/ml}$ standard solution into the separate flasks along with 2 ml of the 5 percent nickel nitrate solution and 10 ml of the 1 percent hydrogen peroxide solution. Dilute to the mark with water. Then treat the standards in the same manner as the samples (Section 4.5.1).

Check these absorbances frequently against 0.8 N HNO_3 (reagent blank) during the analysis to insure that base-line drift has not occurred. Prepare a standard curve of absorbance versus concentration. (Note: For instruments equipped with direct concentration readout devices, preparation of a standard curve will not be necessary.) In all cases, follow calibration and operational

procedures in the manufacturers' instruction manual.

5.2 Sampling Train Calibration. Calibrate the sampling train components according to the indicated Sections of Method 5: Probe Nozzle (Section 5.1), Pitot Tube Assembly (Section 5.2), Metering System (Section 5.3), Probe Heater (Section 5.4), Temperature Gauges (Section 5.5), Leak Check of Metering System (Section 5.6), and Barometer (Section 5.7).

6. Calculations

6.1 Nomenclature—

B_w = Water in the gas stream, proportion by volume.

C_s = Concentration of arsenic as read from the standard curve, $\mu\text{g}/\text{ml}$.

C_a = Actual audit concentration, g/m^3 .

C_d = Determined audit concentration, g/m^3 .

C_i = Arsenic concentration in stack gas, dry basis, converted to standard conditions, g/dsm^3 (g/dscf).

E_a = Arsenic mass emission rate, g/hr .

F_d = Dilution factor (equals 1 if the sample has not been diluted).

I = Percent of isokinetic sampling.

m_b = Total mass of all four impingers and contents before sampling, g .

m_a = Total mass of all four impingers and contents after sampling, g .

m_s = Total mass of arsenic collected in a specific part of the sampling train, μg .

m_i = Total mass of arsenic collected in the sampling train, μg .

T_a = Absolute average dry gas meter temperature (see Figure 108-2), $^{\circ}\text{K}$ ($^{\circ}\text{R}$).

V_a = Volume of gas sample as measured by the dry gas meter, dm^3 (dcf).

$V_{a(\text{std})}$ = Volume of gas sample as measured by the dry gas meter correlated to standard conditions, sm^3 (scf).

V_s = Volume of solution in which the arsenic is contained, ml .

$V_{w(\text{std})}$ = Volume of water vapor collected in the sampling train, corrected to standard conditions, sm^3 (scf).

ΔH = Average pressure differential across the orifice meter (see Figure 108-2), $\text{mm H}_2\text{O}$ (in H_2O).

6.2 Average dry gas meter temperatures (T_a) and average orifice pressure drop (ΔH). See data sheet (Figure 108-2).

6.3 Dry Gas Volume. Using data from this test, calculate $V_{a(\text{std})}$ by using Eq. 5-1 of Method 5. If necessary, adjust the volume for leakages.

6.4 Volume of Water Vapor.

$$V_{w(\text{std})} = K_1 (m_n - m_{n_0}) \quad \text{Eq. 108-1}$$

Where:

$K_1 = 0.001334 \text{ m}^3/\text{g}$ for metric units.

$= 0.047012 \text{ ft}^3/\text{g}$ for English units.

6.5 Moisture Content.

$$B_w = \frac{V_{w(\text{std})}}{V_{m(\text{std})} + V_{w(\text{std})}} \quad \text{Eq. 108-2}$$

6.6 Amount of arsenic collected.

6.6.1 Calculate the amount of arsenic collected in each part of sampling train, as follows:

$$m_n = C_s F_d V_n \quad \text{Eq. 108-3}$$

6.6.2 Calculate the total amount of arsenic collected in the sampling train as follows:

$$m_i = m_n(\text{filters}) + m_n(\text{probe}) + m_n(\text{impingers}) - m_n(\text{filter blank}) - m_n(\text{NaOH}) - m_n(\text{H}_2\text{O}) \quad \text{Eq. 108-4}$$

6.7 Calculate the arsenic concentration in the stack gas (dry basis, adjusted to standard conditions) as follows:

$$C_i = K_2 (m_i / V_{a(\text{std})}) \quad \text{Eq. 108-5}$$

Where:

$$K_2 = 10^{-6} \text{ g}/\mu\text{g}$$

6.8 Pollutant Mass Rate. Calculate the arsenic mass emission rate using the following equation.

$$E_a = C_i Q_{a(\text{std})} \quad \text{Eq. 108-6}$$

The volumetric flow rate, $Q_{a(\text{std})}$, should be calculated as indicated in Method 2.

6.9 Isokinetic Variation. Using data from this test, calculate I . Use Eq. 5-8 of Method 5.

6.10 Acceptable Results. Same as Method 5, Section 6.12.

6.11 Relative Error (RE) for QA Audits, Percent.

$$\text{RE} = \frac{C_a - C_c}{C_c} \times 100 \quad \text{Eq. 108-7}$$

7. Bibliography

1. Same as Citations 1 through 9 of Section 7, of Method 5.

2. Perkin Elmer Corporation. Analytical Methods for Atomic Absorption Spectrophotometry. 303-0152. Norwalk, Connecticut. September 1976. pp. 5-6.

3. Standard Specification for Reagent Water. In: Annual Book of American Society for Testing and Materials Standards. Part 31: Water, Atmospheric Analysis. American Society for Testing and Materials. Philadelphia, PA. 1974. pp. 40-42.

METHOD 108A—DETERMINATION OF ARSENIC CONTENT IN ORE SAMPLES FROM NONFERROUS SMELTERS

1. Applicability and Principle

1.1 Applicability. This method applies to the determination of inorganic arsenic (As) content of process ore and reverberatory matte samples from nonferrous smelters and other sources as specified in the regulations.

1.2 Principle. Arsenic bound in ore samples is liberated by acid digestion and analyzed by atomic absorption spectrophotometry.

2. Apparatus

2.1 Sample Preparation

2.1.1 Parr Acid Digestion Bomb. Stainless steel with vapor-tight Teflon cup and cover.

2.1.2 Volumetric Pipets. 2- and 5-ml sizes.

2.1.3 Volumetric Flask. 50-ml polypropylene with screw caps, (one needed per standard).

2.1.4 Funnel. Polyethylene or polypropylene.

2.1.5 Oven. Capable of maintaining a temperature of approximately 105°C.

2.1.6 Analytical Balance. To measure to within 0.1 mg.

2.2 Analysis.

2.2.1 Spectrophotometer and Recorder. Equipped with an electrodeless discharge lamp and a background corrector to measure absorbance at 193.7 nm. A graphite furnace may be used in place of the vapor generator accessory when measuring samples with low As levels. The recorder shall match the output of the spectrophotometer.

2.2.2 Volumetric Flasks. Class A, 50-ml (one needed per sample and blank).

2.2.3 Volumetric Pipets. Class A, 1-, 5-, 10-, and 25-ml sizes.

3. Reagents

Unless otherwise specified, use ACS reagent grade (or equivalent) chemicals throughout.

3.1 Sample Preparation.

3.1.1 Water. Deionized distilled to meet American Society for Testing and Materials Specification D-1193-74, Type 3 (incorporated by reference—See § 60.7). When high concentrations of organic matter are not expected to be present, the analyst may omit the $\text{K}_2\text{Cr}_2\text{O}_7$ test for oxidizable organic matter. Use in all dilutions requiring water.

3.1.2 Nitric Acid (HNO_3), Concentrated. **HANDLE WITH CAUTION.**

3.1.3 Nitric Acid, 0.5 N. In a 1-liter volumetric flask containing water, add 32 ml of concentrated HNO_3 and dilute to volume with water.

3.1.4 Hydrofluoric Acid (HF), Concentrated. **HANDLE WITH CAUTION.**

3.1.5 Potassium Chloride (KCl) Solution, 10 percent (w/v). Dissolve 10 g KCl in water,

add 3 ml concentrated HNO_3 , and dilute to 100 ml.

3.1.6 Filter. Teflon filters, 3 micron porosity, 47mm size. (Available from Millipore Co., type FS, Catalog Number FSLW04700).

3.1.7 Sodium Borohydride (NaBH_4), 5 Percent (W/V). Dissolve 5.00 g of NaBH_4 in about 500 ml of 0.1 NaOH in a 1-liter volumetric flask. Then, dilute to exactly 1.0 liter with 0.1 NaOH .

3.1.8 Nickel Nitrate, 5 Percent (W/V). Dissolve 24.780 g of nickel nitrate hexahydrate in water in a 100-ml volumetric flask and dilute to 100 ml with water.

3.1.9 Nickel Nitrate, 1 percent (W/V). Pipet 20 ml of 5 percent nickel nitrate solution into a 100-ml volumetric flask and dilute to 100 ml with water.

3.2 Analysis.

3.2.1 Water. Same as in Section 3.1.1.

3.2.2 Sodium Hydroxide (NaOH), 0.1 N. Dissolve 2.00 g of NaOH in water in a 500-ml volumetric flask. Dilute to volume with water.

3.2.3 Nitric Acid, 0.5 N. Same as in Section 3.1.3.

3.2.4 Potassium Chloride Solution, 10 percent. Same as in Section 3.1.5.

3.2.5 Stock Arsenic Standard, 1 mg As/ml. Dissolve 1.320 g of primary grade As_2O_3 in 20 ml of 0.1 N NaOH . Slowly add 30 ml of concentrated HNO_3 , and heat in an oven at 105°C for 2 hours. Allow to cool, and dilute to 1 liter with deionized distilled water.

3.2.6 Nitrous Oxide. Suitable quality for atomic absorption analysis.

3.2.7 Acetylene. Suitable quality for atomic absorption analysis.

3.2.8 Quality Assurance Audit Samples. Arsenic samples prepared by the Environmental Protection Agency's (EPA) Environmental Systems Laboratory, Quality Assurance Division, Source Branch, Mail Drop 77A, Research Triangle Park, North Carolina 27711. Each set will consist of two vials of unknown concentrations. Only when making compliance determinations, obtain an audit sample set from the Quality Assurance Management Office at each EPA regional office or the responsible enforcement office. (NOTE: The tester should notify the Quality Assurance Office or the responsible enforcement agency at least 30 days prior to the test date to allow sufficient time for delivery.)

4. Procedure

4.1 Sample Collection. A sample that is representative of the ore lot to be tested must be taken prior to analysis. The sample must be ground into a finely pulverized state. (A portion of the samples routinely collected for metals analysis may be used provided the sample is representative of the ore being tested.)

4.2 Sample Preparation. Weigh 50 to 500 mg of finely pulverized sample to the nearest 0.1 mg. Transfer the sample into the Teflon cup of the digestion bomb, and add 2 ml each of concentrated HNO_3 and HF . Seal the bomb immediately to prevent the loss of any volatile arsenic compounds that may form. Heat in an oven 105°C for 2 hours. Then remove the bomb from the oven and allow it to cool. Using a Teflon filter, quantitatively filter the digested sample into a 50-ml polypropylene volumetric flask. Rinse the bomb three times with small portions of 0.5 N HNO_3 , and filter the rinses into the flask. Add 5 ml of KCl solution to the flask, and dilute to 50 ml with 0.5 N HNO_3 .

4.3 Spectrophotometer Preparation. Turn on the power; set the wavelength, slit width, and lamp current; and adjust the background corrector as instructed by the manufacturer's manual for the particular atomic absorption spectrophotometer. Adjust the burner and flame characteristics as necessary.

4.4 Preparation of Standard Solutions. Pipet 1, 5, 10, and 25 ml of the stock As solution into separate 100-ml volumetric flasks. Add 10 ml KCl solution and dilute to the mark with 0.5 N HNO_3 . This will give standard concentrations of 10, 50, 100, and 250 μg As/ml. For low-level-arsenic samples that require the use of a graphite furnace or vapor generator, follow the procedures in Section 4.4.1.

Dilute 10 ml of KCl solution to 100 ml with 0.5 N HNO_3 , and use as a reagent blank. Measure the standard absorbances against the reagent blank. Check these absorbances frequently against the blank during the analysis to assure that baseline drift has not occurred.

Prepare a standard curve of absorbance versus concentration. (Note: For instruments equipped with direct concentration readout devices, preparation of a standard curve will not be necessary.) In all cases follow calibration and operational procedures in the manufacturer's instruction manual. Maintain a laboratory log of all calibrations.

4.4.1 Arsenic Determination at Low Concentration. The lower limit of flame atomic absorption spectrophotometry is 10 μg As/ml. If the arsenic concentration of any sample is at a lower level, use the vapor generator or graphite furnace which is available as an accessory component. Follow the manufacturer's instructions in the use of such equipment.

4.4.1.1 Vapor Generator Procedure. Place a sample containing between 0 and 5 μg of arsenic in the reaction tube, and dilute to 15 ml with water. Since there is some trial and error involved in this procedure, it may be necessary to screen the samples by conventional atomic absorption until an approximate concentration is determined. After de-

termining the approximate concentration, adjust the volume of the sample accordingly. Pipet 15 ml of concentrated HCl into each tube. Add 1 ml of 30 percent KI solution. Place the reaction tube into a 50°C water bath for 5 minutes. Cool to room temperature. Connect the reaction tube to the vapor generator assembly. When the instrument response has returned to baseline, inject 5.0 ml of 5 percent NaBH_4 , and integrate the resulting spectrophotometer signal over a 30-second time period.

4.4.1.2 Graphite Furnace Procedure. Pipet 5 ml of this digested solution into a 10-ml volumetric flask. Add 1 ml of the 1 percent nickel nitrate solution, 0.5 ml of 50 percent HNO_3 , and 1 ml of the 3 percent hydrogen peroxide and dilute to 10 ml with water. The sample is now ready to inject in the furnace for analysis.

Because instruments from different manufacturers are different, no detailed operating instructions are given here. Instead, the analyst should follow the instructions provided with the particular instrument.

4.5 Analysis.

4.5.1 Arsenic Determination. Determine the absorbance of each sample using the blank as a reference. If the sample concentration falls outside the range of the calibration curve, make an appropriate dilution with 0.5 N HNO_3 , so that the final concentration falls within the range of the curve. From the curve, determine the As concentration in each sample.

4.5.2 Mandatory Check for Matrix Effects on the Arsenic Results. Same as in Method 12, Section 5.4.2.

4.5.3 Audit analysis. With each set or sets of source compliance samples, analyze the two unknown audit samples in the same manner as the source samples to evaluate the techniques of the analyst and the standards preparation. The same analyst, analytical reagents, and analytical system shall be used both for each set or sets of compliance samples and the EPA audit samples; if this condition is met, it is not necessary to analyze additional audit samples for subsequent compliance analyses performed for the same enforcement agency within a 30-day period. An audit sample set may not be used to validate different sets of compliance samples under the jurisdiction of different enforcement agencies unless prior arrangements are made with both enforcement agencies.

Calculate the concentration in g/m^3 using the specified sample volume in the audit instructions. (Note: The acceptability of the analyses of the audit samples may be obtained immediately by reporting the audit and compliance results by telephone). Include the results of both audit samples, their identification numbers, and the analysts' names with the results of the compliance determination samples in appropriate

reports to the EPA regional office or the appropriate enforcement agency. Include this information with subsequent compliance analyses for the same enforcement agency during the succeeding 30-day period.

5. Calculations

5.1 Calculate the percent arsenic in the ore sample as follows:

$$\% \text{ AS} = \frac{5 C_s F_d}{W} \quad \text{Eq. 108A-1}$$

Where:

C_s = Concentration of As as read from the standard curve, $\mu\text{g/ml}$.

F_d = Dilution factor (equals 1 if the sample has not been diluted).

W = Weight of ore sample analyzed, mg.
 $5 = (50 \text{ ml sample} \times 100) / (10^3 \mu\text{g/mg})$.

6. Bibliography

1. Same as Citations 1 through 9 of Section 7, of Method 5.

2. Perkin Elmer Corporation. Analytical methods of Atomic Absorption Spectrophotometry. 303-0152. Norwalk, Connecticut. September 1976. pp 5-6.

3. Ringwald, D. (TRW). Arsenic Determination on Process Materials from ASARCO's Copper Smelter in Tacoma, Washington. Unpublished Report. Prepared for Emission Measurement Branch, Emission Standards and Engineering Division, U.S. Environmental Protection Agency, Research Triangle Park, North Carolina 27711. August 1980. 35 p.

METHOD 108B—DETERMINATION OF ARSENIC CONTENT IN ORE SAMPLES FROM NONFERROUS SMELTERS

1. Applicability and Principle

1.1 Applicability. This method applies to the determination of inorganic arsenic (As) content of process ore and reverberatory matte samples from nonferrous smelters and other sources as specified in the regulations. Samples resulting in an analytical concentration greater than $10 \mu\text{g As/ml}$ may be analyzed by this method.

1.2 Principle. Arsenic bound in ore samples is liberated by acid digestion and analyzed by flame atomic absorption spectrophotometry.

2. Apparatus

2.1 Sample Preparation.

2.2.1 Teflon Beakers. 150-ml.

2.1.2 Graduated Pipets. 5-ml disposable.

2.1.3 Graduated Cylinder. 50-ml.

2.1.4 Volumetric Flask. 100-ml.

2.1.5 Analytical Balance. To measure within 0.1 mg.

2.1.6 Hot Plate.

2.1.7 Perchloric Acid Fume Hood.

2.2 Analysis.

2.2.1 Spectrophotometer. Equipped with an electrodeless discharge lamp and a background corrector to measure absorbance at 193.7 nm.

2.2.2 Beaker and Watch Glass. 400-ml.

2.2.3 Volumetric Flask. 1-liter.

2.2.4 Volumetric Pipets. 1-, 5-, 10-, and 25 ml.

3. Reagents

Unless otherwise specified, use American Chemical Society (ACS) reagent grade (or equivalent) chemicals throughout.

3.1 Sample Preparation.

3.1.1 Water. Deionized distilled to meet American Society for Testing and Materials Specification D 1193-74, Type 3 (incorporated by reference—see § 61.18).

3.1.2 Nitric Acid (HNO_3). Concentrated. HANDLE WITH CAUTION.

3.1.3 Hydrofluoric Acid (HF). Concentrated. HANDLE WITH CAUTION.

3.1.4 Perchloric Acid (HClO_4). 70 Percent. HANDLE WITH CAUTION.

NOTE: Because of its caustic, hygroscopic, and deflagrating nature, use extreme care in handling HClO_4 . Keep separate from water and oxidizable materials to prevent vigorous evolution of heat, spontaneous combustion, or explosion. Heat solutions containing HClO_4 only in hoods specifically designed for HClO_4 .

3.1.5 Hydrochloric Acid (HCl). Concentrated. HANDLE WITH CAUTION.

3.2 Analysis.

3.2.1 Water. Same as in Section 3.1.1.

3.2.2 Stock Arsenic Standard. 1.0 mg As/ml. Dissolve 1.3203 g of primary grade As_2O_3 (dried at 105°C) in a 400-ml beaker with 10 ml of HNO_3 and 5 ml HCl . Cover with a watch glass and heat gently until dissolution is complete. Add 10 ml of HNO_3 and 15 ml of HClO_4 , evaporate to strong fumes of HClO_4 , and reduce to about 20 ml volume. Cool, add 100 ml of water and 100 ml of HCl , and transfer quantitatively to a 1-liter volumetric flask. Dilute to volume with water and mix.

3.2.3 Acetylene. Suitable quality for atomic absorption analysis.

3.2.4 Air. Suitable quality for atomic absorption analysis.

3.2.5 Quality Assurance Audit Samples. Same as in Method 108A, Section 3.2.8.

4. Procedure

4.1 Sample Collection. Same as in Method 108A, Section 4.1.

4.2 Sample Preparation. Weigh 100 to 1000 mg of finely pulverized sample to the nearest 0.1 mg. Transfer the sample to a 150-ml Teflon beaker. Dissolve the sample by adding 15 ml of HNO_3 , 10 ml of HCl , 10 ml of HF, and 10 ml of HClO_4 , in the exact

order as described, and let stand for 10 minutes. In a HClO_4 fume hood, heat on a hot plate until 2-3 ml of HClO_4 remain, then cool. Add 20 ml of water and 10 ml of HCl . Cover and warm until the soluble salts are in solution. Cool, and transfer quantitatively to 100-ml volumetric flask. Dilute to the mark with water.

4.3 Spectrophotometer Preparation. Same as in Method 108A, Section 4.3.

4.4 Preparation of Standard Solutions.

4.4.1 Pipet 1, 5, 10, and 25 ml of the stock As solution into separate 100-ml flasks. Add 2 ml of HClO_4 , 10 ml of HCl , and dilute to the mark with water. This will provide standard concentrations of 10, 50, 100, and 250 $\mu\text{g As/ml}$. For lower level arsenic samples, use Method 108C.

4.4.2 Measure the standard absorbances against the reagent blank. Check these absorbances frequently against the blank during the analysis to ensure that baseline drift has not occurred.

4.4.3 Prepare a standard curve of absorbance versus concentration.

NOTE.—For instruments equipped with direct concentration readout devices, preparation of a standard curve will not be necessary. In all cases, follow calibration and operational procedures in the manufacturer's instruction manual. Maintain a laboratory log of all calibrations.

4.5 Analysis.

4.5.1 Arsenic Determination. Determine the absorbance of each sample using the blank as a reference. If the sample concentration falls outside the range of the calibration curve, make an appropriate dilution with 2 percent HClO_4 /10 percent HCl (prepared by diluting 2 ml concentrated HClO_4 and 10 ml concentrated HCl to 100 ml with water) so that the final concentration falls within the range of the curve. From the curve, determine the As concentration in each sample.

4.5.2 Mandatory Check for Matrix Effects on the Arsenic Results. Same as in Method 12, Section 5.4.2, 40 CFR part 60.

4.5.3 Audit Analysis. Same as in Method 108A, Section 4.5.3.

5. Calculations

Same as in Method 108A, Section 5.

6. Bibliography

Same as in Method 108A, Section 6.

METHOD 108C—DETERMINATION OF ARSENIC CONTENT IN ORE SAMPLES FROM NONFERROUS SMELTERS

1. Applicability and Principle

1.1 Applicability. This method applies to the determination of inorganic arsenic (As) content of process ore and reverberatory matte samples from nonferrous smelters and other sources as specified in the regulations. This method is applicable to samples having an analytical concentration less than 10 $\mu\text{g As/ml}$.

1.2 Principle. Arsenic bound in ore samples is liberated by acid digestion and analyzed by the molybdenum blue photometric procedure.

2. Apparatus

2.1 Sample Preparation and Distillation.

2.1.1 Analytical Balance. To measure to within 0.1 mg.

2.1.2 Erlenmeyer Flask. 300-ml.

2.1.3 Hot Plate.

2.1.4 Distillation Apparatus. No. 6, American Society for Testing and Materials (ASTM) E50, reapproved 1986 (incorporated by reference—see § 60.18); detailed in Figure 108C-1.

2.1.5 Graduated Cylinder. 50-ml.

2.1.6 Perchloric Acid Fume Hood.

2.2 Analysis.

2.2.1 Photometer. Capable of measuring at 660 nm.

2.2.2 Volumetric Flasks. 50- and 100-ml.

3. Reagents

Unless otherwise specified, use ACS reagent grade (or equivalent chemicals) throughout.

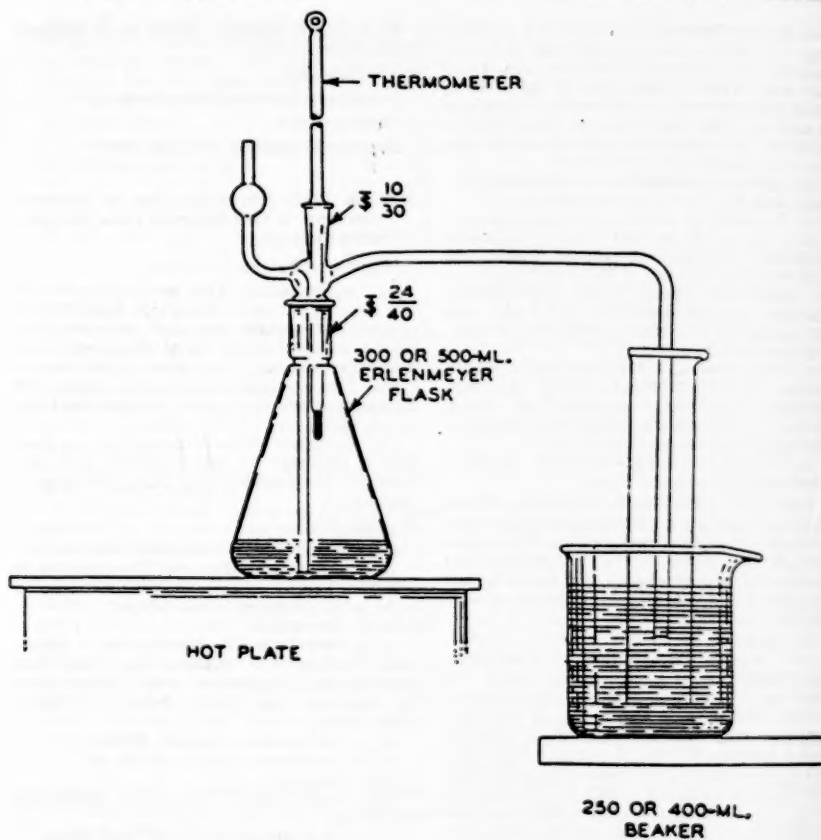


Figure 108C-1. Distillation apparatus.

11a

3.1 Sample Preparation.

3.1.1 Water. Deionized distilled to meet ASTM Specification D 1193-74, Type 3 (incorporated by reference—see § 61.18). When high concentrations of organic matter are not expected to be present, the analyst may omit the KMnO_4 test for oxidizable organic matter.

3.1.2 Nitric Acid (HNO_3), Concentrated. HANDLE WITH CAUTION.

3.1.3 Hydrofluoric Acid (HF), Concentrated. HANDLE WITH CAUTION.

3.1.4 Sulfuric Acid (H_2SO_4), Concentrated. HANDLE WITH CAUTION.

3.1.5 Perchloric Acid (HClO_4), 70 Percent. HANDLE WITH CAUTION.

NOTE.—Because of its caustic, hygroscopic, and deflagrating nature, use extreme care in handling HClO_4 . Keep separate from water and oxidizable materials to prevent vigorous evolution of heat, spontaneous combustion, or explosion. Heat solutions containing HClO_4 only in hoods specifically designed for HClO_4 .

3.1.6 Hydrochloric Acid (HCl), Concentrated. HANDLE WITH CAUTION.

3.1.7 Dilute Hydrochloric Acid. Add one part concentration HCl to nine parts water.

3.1.8 Hydrazine Sulfate [$(\text{NH}_2)_2\text{H}_2\text{SO}_4$].

3.1.9 Potassium Bromide (KBr).

3.1.10 Bromine Water, Saturated.

3.2 Analysis.

3.2.1 Water. Same as in Section 3.1.1.

3.2.2 Methyl Orange Solution, 1 g/liter.

3.2.3 Ammonium Molybdate Solution, 5 g/liter. Dissolve 0.5 g ($(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}$) in water in a 100-ml volumetric flask, and dilute to the mark. This solution shall be freshly prepared.

3.2.4 Standard Arsenic Solution, 10 μg As/ml. Dissolve 0.1320 g of As_2O_3 in 100 ml HCl in a 1-liter volumetric flask. Add 200 ml of water, cool, dilute to the mark with water, and mix. Transfer 100 ml of this solution to a 1-liter volumetric flask, add 40 ml HCl , cool, dilute to the mark, and mix.

3.2.5 Hydrazine Sulfate Solution, 1 g/liter. Dissolve 0.1 g of $(\text{NH}_2)_2\text{H}_2\text{SO}_4$ in water, and dilute to 100 ml in a volumetric flask. This solution shall be freshly prepared.

3.2.6 Potassium Bromate (KBrO_3) Solution, 0.03 Percent. Dissolve 0.3 g KBrO_3 in water, and dilute to 1 liter with water.

3.2.7 Ammonium Hydroxide (NH_4OH), Concentrated.

3.2.8 Boiling Granules.

3.2.9 1/1 HCl /Water. Dilute equal parts concentrated HCl with water.

3.2.10 Quality Assurance Audit Samples. Same as in Method 108A, Section 3.2.8.

4. Procedure

4.1 Sample Preparation and Distillation.

4.1.1 Weigh 1.0 g of finely pulverized sample to the nearest 0.1 mg. Transfer the sample to a 300-ml Erlenmeyer flask and add 15 ml of HNO_3 , 4 ml HCl , 2 ml HF , 3 ml

HClO_4 , and 15 ml H_2SO_4 . In a HClO_4 fume hood, heat on a hot plate to decompose the sample. Then heat while swirling over an open flame until dense, white fumes evolve. Cool, add 15 ml of water, swirl to hydrate the H_2SO_4 completely, and add several boiling granules. Cool to room temperature.

4.1.2 Add 1 g of KBr , 1 g hydrazine sulfate, and 50 ml HCl . Immediately attach the distillation head with thermometer and dip the side arm into a 50-ml graduated cylinder containing 25 ml of water and 2 ml of bromine water. Keep the graduated cylinder immersed in a beaker of cold water during distillation. Distill until the temperature of the vapor in the flask reaches 107°C . When distillation is complete, remove the flask from the hot plate, and simultaneously wash down the side arm with water as it is removed from the cylinder.

4.1.3 If the expected arsenic content is in the range of 0.0020 to 0.10 percent, dilute the distillate to the 50-ml mark of the cylinder with water, stopper, and mix. Transfer a 5.0-ml aliquot to a 50-ml volumetric flask. Add 10 ml of water and a boiling granule. Place the flask on a hot plate, and heat gently until the bromine is expelled and the color of methyl orange indicator persists upon the addition of 1-2 drops. Cool the flask to room temperature. Neutralize just to the yellow color of the indicator with dropwise additions of NH_4OH . Bring back to the red color by dropwise addition of dilute HCl , and add 10 ml excess. Proceed with the molybdenum blue color development as described in section 4.2.

4.1.4 If the expected arsenic content is in the range of 0.0002 to 0.0010 percent As, transfer either the entire initial distillate or the measured remaining distillate from above to a 250-ml beaker. Wash the cylinder with two successive portions of concentrated HNO_3 , adding each portion to the distillate in the beaker. Add 4 ml of concentrated HClO_4 , a boiling granule, and cover with a flat watch glass placed slightly to one side. Boil gently on a hot plate until the volume is reduced to approximately 10 ml. Add 3 ml of HNO_3 , and continue the evaporation until HClO_4 is refluxing on the beaker cover. Cool briefly, rinse the underside of the watch glass and the inside of the beaker with about 3-5 ml of water, cover, and continue the evaporation to expel all but 2 ml of the HClO_4 . NOTE: If the solution appears cloudy due to a small amount of antimony distilling over, add 4 ml of 1/1 HCl /water and 5 ml of water, cover, and warm gently until clear. If cloudiness persists, add 5 ml of HNO_3 and 2 ml H_2SO_4 . Continue the evaporation of the volatile acids to solubilize the antimony until dense white fumes of H_2SO_4 appear. Retain at least 1 ml of the H_2SO_4 . To the 2 ml of HClO_4 solution or 1 ml of the H_2SO_4 solution, add 15 ml of

water, boil gently for 2 minutes, and then cool. Proceed with the molybdenum blue color development by neutralizing the solution directly in the beaker just to the yellow indicator color by dropwise addition of NH_4OH . Just bring back the red color by dropwise addition of dilute HCl . Transfer the solution to a 50-ml volumetric flask, and rinse the beaker successively with 10 ml of dilute HCl , followed by several small portions of water. At this point the volume of solution in the flask should be no more than 40 ml. Continue with the color development as described in section 4.2.

4.2 Analysis.

4.2.1 Add 1 ml of KBrO_3 solution to the flask and heat on a low-temperature hot plate to about 50°C to oxidize the arsenic and methyl orange. Add 5.0 ml of ammonium molybdate solution to the warm solution and mix. Add 2.0 ml of hydrazine sulfate solution, dilute until the solution comes within the neck of the flask, and mix. Place in a 400-ml beaker, 30 percent full of boiling water, for 10 minutes. Enough heat must be supplied to prevent the water bath from cooling much below the boiling point upon inserting the volumetric flask. Remove the flask, cool to room temperature, dilute to the mark, and mix.

4.2.2 Transfer a suitable portion of the reference solution to an absorption cell, and adjust the photometer to the initial setting, using a light band centered at 660 nm. While maintaining this photometer adjustment, take the photometric readings of the calibration solutions followed by the samples.

4.3 Preparation of the Calibration Curve. Transfer 1.0, 2.0, 4.0, 8.0, 12.0, 16.0, and 20.0 ml of standard arsenic solution ($10\text{ }\mu\text{g/ml}$) to each of the seven 50-ml volumetric flasks. Dilute to 20 ml with dilute HCl . Add one drop of methyl orange solution and neutralize to the yellow color with dropwise addition of NH_4OH . Just bring back to the red color by dropwise addition of dilute HCl , and add 10 ml in excess. Proceed with the color development as described in section 4.2. Plot the photometric readings of the calibration solutions against $\mu\text{g As per 50 ml}$ of solution. From the curve, determine the As concentration in each sample.

4.4 Audit Analysis. Same as in Method 108A, section 4.5.3.

5. Calculations

Same as in Method 108A, section 5.

6. Bibliography

Ringwald, D. (TRW). Arsenic Determination on Process Materials from ASARCO's Copper Smelter in Tacoma, Washington. Unpublished Report. Prepared for the Emission Measurement Branch, Technical Support Division, U.S. Environmental Protection Agency, Research Triangle Park, North Carolina 27711. August 1980. 35 p.

METHOD 111—DETERMINATION OF POLONIUM-210 EMISSIONS FROM STATIONARY SOURCES

Performance of this method should not be attempted by persons unfamiliar with the use of equipment for measuring radioactive disintegration rates.

1.0 Applicability and Principle

1.1 Applicability. This method is applicable to the determination of polonium-210 emissions in particulate samples collected in stack gases. Samples should be analyzed within 30 days of collection to minimize error due to growth of polonium-210 from any lead-210 present in the sample.

1.2 Principle A particulate sample is collected from stack gases as described in Method 5 of Appendix A to 40 CFR Part 60. The polonium-210 in the sample is put in solution, deposited on a metal disc, and the radioactive disintegration rate measured. Polonium in acid solution spontaneously deposits on surfaces of metals that are more electropositive than polonium. This principle is routinely used in the radiochemical analysis of polonium-210.

2.0 Apparatus

2.1 Alpha spectrometry system consisting of a multichannel analyzer, biasing electronics, silicon surface barrier detector, vacuum pump and chamber.

2.2 Constant temperature bath at 85°C .

2.3 Polished silver discs, 3.8 cm diameter, 0.4 mm thick with a small hole near the edge.

2.4 Glass beakers, 400 ml, 150 ml.

2.5 Hot plate, electric.

2.6 Fume hood.

2.7 Teflon beakers, 150 ml.

2.8 Magnetic stirrer.

2.9 Stirring bar.

2.10 Plastic or glass hooks to suspend plating discs.

2.11 Internal proportional counter for measuring alpha particles.

2.12 Nucleopore filter membranes, 25 mm diameter, 0.2 micrometer pore size or equivalent.

2.13 Planchets, stainless steel, 32 mm diameter with 1.5 mm lip.

2.14 Transparent plastic tape, 2.5 cm wide with adhesive on both sides.

2.15 Epoxy spray enamel.

2.16 Suction filter apparatus for 25 mm diameter filter.

2.17 Wash bottles, 250 ml capacity.

2.18 Plastic graduated cylinder, 25 ml capacity.

3.0 Reagents

3.1 Ascorbic acid, Reagent grade.

3.2 Ammonium hydroxide (NH_4OH) 15 M, Reagent grade.

3.3 Distilled water meeting ASTM specifications for Type 3 Reagent Water. ASTM

Test Method D 1193-77 (incorporated by reference-Section 61.18).

3.4 Ethanol (C_2H_5OH), 95 percent, Reagent grade.

3.5 Hydrochloric acid (HCl), 12 M, Reagent grade.

3.6 Hydrochloric acid, 1 M, dilute 83 ml of the 12 M Reagent grade HCl to 1 liter with distilled water.

3.7 Hydrofluoric acid (HF), 29 M, Reagent grade.

3.8 Hydrofluoric acid, 3 M, dilute 52 ml of the 29 M Reagent grade HF to 500 ml with distilled water. Use a plastic graduated cylinder and storage bottle.

3.9 Lanthanum carrier, 0.1 mg La^{+3}/ml . Dissolve 0.078 gram Reagent grade lanthanum nitrate, $La(NO_3)_3 \cdot 6H_2O$ in 250 ml of 1 M HCl.

3.10 Nitric acid (HNO_3), 16 M, Reagent grade.

3.11 Perchloric acid ($HClO_4$), 12 M, Reagent grade.

3.12 Polonium-209 solution.

3.13 Commercial silver cleaner.

3.14 Degreaser.

3.15 Standard solution of plutonium or americium.

3.16 Volumetric flask, 100 ml, 250 ml.

4.0 Procedure

4.1 Sample Preparation.

The glass fiber filter and acetone rinse from Method 5 of Appendix A to 40 CFR part 60 are combined and dissolved as described below.

4.1.1 Add polonium-209 tracer to the acetone rinse in the glass beaker from Method 5 in an amount approximately equal to the amount of polonium-210 expected in the total particulate sample. Add 16 M nitric acid to the beaker to digest and loosen the residue.

4.1.2 Transfer the residue from the glass beaker to a teflon beaker containing the glass fiber filter. Rinse the glass beaker with 16 M nitric acid. If necessary reduce the volume in the beaker by evaporation until all of the nitric acid from the glass beaker has been transferred to the teflon beaker.

4.1.3 Add 30 ml of 29 M hydrofluoric acid to the teflon beaker and evaporate to near dryness on a hot plate in a properly operating hood. Caution: Do not allow the residue to go to dryness and overheat; this will result in loss of polonium.

4.1.4 Repeat step 4.1.3 until filter is dissolved.

4.1.5 Add 100 ml of 16 M nitric acid to the residue in the teflon beaker and evapo-

rate to near dryness. Caution: Do not allow the residue to go to dryness.

4.1.6 Add 50 ml of 16 M nitric acid and 10 ml of 12 M perchloric acid to the teflon beaker and heat until dense fumes of perchloric acid are evolved.

4.1.7 Repeat steps 4.1.3 to 4.1.6 as necessary until sample is completely dissolved.

4.1.8 Add 10 ml of 12 M hydrochloric acid and evaporate to dryness. Repeat additions and evaporations several times.

4.1.9 Transfer the sample to a 250 ml volumetric flask and dilute to volume with 3 M hydrochloric acid.

4.2 Sample Screening.

The samples are checked for radioactivity levels to avoid contamination of the alpha spectrometry system. Use the following screening method:

4.2.1 Twenty ml of 1 M HCl are added to a 150 ml beaker.

4.2.2 One ml of the lanthanum carrier solution, 0.1 mg lanthanum per ml, is added to beaker.

4.2.3 A 1 ml aliquot of solution from Section 4.1.7 is added to the beaker.

4.2.4 Three ml of 15 M ammonium hydroxide are added to the beaker.

4.2.5 The solution from Section 4.2.4 is allowed to stand for a minimum of 30 minutes.

4.2.6 The solution is filtered through a filter membrane using suction.

4.2.7 The membrane is washed with 10 ml of distilled water and 5 ml of ethanol.

4.2.8 The membrane is allowed to air dry and then mounted, filtration side up, on a planchet lined with double-side plastic tape.

4.2.9 The membrane is radioassayed using an internal proportional alpha counter.

4.2.10 The activity of the original solution from Section 4.1.7 is calculated using Eq. 111-1.

$$P = \frac{250 C_s - C_B}{2.22 E_1 A_1 T} \quad \text{Eq. 111-1}$$

where:

P = total activity of original solution from Section 4.1.7, in pCi.

C_s = total counts of screening sample.

C_B = total counts of procedure background. (See 4.6).

E_1 = counting efficiency as determined in Section 8.0, counts per minute per disintegration per minute.

2.22 = disintegrations per minute per picocurie.

A_1 = aliquot used in Section 4.2.3 in ml if different from 1 ml.

¹Mention of registered trade names or specific products does not constitute endorsement by the Environmental Protection Agency.

T=counting time in minutes for sample and background (which must be equal).

250=volume of solution from Section 4.1.7 in ml.

4.2.11 Determine the aliquot volume of solution from Section 4.1.7 to be analyzed for polonium-210 using results of the calculation described in Section 4.2.10. The aliquot used should contain an activity between 1 and 4 picocuries.

$$A_s = \frac{250 \text{ (desired picocuries in aliquot)}}{P}$$

Eq. 111-2

A_s =aliquot to be analyzed in ml.

P=total activity, as calculated with Eq. 111-1.

4.3 Preparation of Silver Disc for Spontaneous Electrodeposition.

4.3.1 Clean both sides of disc with a mild abrasive commercial silver cleaner.

4.3.2 Clean both sides of disc with degreaser.

4.3.3 Place disc on absorbent paper and spray one side with epoxy spray enamel. This should be carried out in a well-ventilated area, with the disc lying flat to keep paint on one side only.

4.3.4 Allow paint to dry for 24 hours before using disc for deposition.

4.4 Sample Analysis.

4.4.1 Add the aliquot of solution from Section 4.1.7 to be analyzed as determined in Section 4.2.11 to a suitable 200 ml container to be placed in a constant temperature bath. Note, aliquot volume may require a larger container.

4.4.2 If necessary, bring the volume to 100 ml with 1 M HCl. If the aliquot volume exceeds 100 ml, use total aliquot.

4.4.3 Add 200 mg of ascorbic acid and heat solution to 85 °C in a constant temperature bath.

4.4.4 Stirring of the solution must be maintained while the solution is in the constant temperature bath for plating.

4.4.5 Suspend a silver disc in the heated solution using a glass or plastic rod with a hook inserted through the hole in the disc. The disc should be totally immersed in the solution at all times.

4.4.6 Maintain the disc in solution for 3 hours while stirring.

4.4.7 Remove the silver disc, rinse with distilled water and allow to air dry at room temperature.

4.5 Measurement of Polonium-210

4.5.1 Place the silver disc, with deposition side (unpainted side) up, on a planchet and secure with double-side plastic tape.

4.5.2 Place the planchet with disc in alpha spectrometry system and count for 1000 minutes.

4.6 Determination of Procedure Background.

Background counts used in all equations are determined by performing the specific analysis required using the analytical reagents only. This should be repeated every 10 analyses.

4.7 Determination of Instrument Background.

Instrument backgrounds of the internal proportional counter and alpha spectrometry system should be determined on a weekly basis. Instrument background should not exceed procedure background. If this occurs, it may be due to a malfunction or contamination.

5.0 Calculation of Polonium-210 Activity

5.1 Calculate the activity of polonium-210 on a sample filter using Eq. 111-3

$$A = \frac{C_T - C_B L}{2.22 E_v E_c T D} \quad \text{Eq. 111-3}$$

where:

A=picocuries of polonium-210 in the particulate sample.

C_T =total counts in polonium-210 spectral region.

C_B =procedure background counts in polonium-210 spectral region.

L=dilution factor. This is the volume in ml of solution in Section 4.1.7 (250 ml) divided by volume in ml used in Section 4.4.1.

2.22=disintegrations per minute per picocurie.

E_v =fraction of polonium recovered on the planchet. Given by:

$$E_v = \frac{B_T - B_B}{2.22 F E_c T}$$

where:

B_T =polonium-209 tracer counts in sample.

B_B =procedure background counts measured in polonium-209 spectral region.

F=activity in picocuries of polonium-209 added to sample—from Eq. 111-7.

2.22=disintegrations per minute per picocurie.

E_c =counting efficiency of detector used, given by Eq. 111-6, as counts per minute per disintegration per minute.

T=counting time, specified in Sections 4.5.1 and 7.11 as 1000 minutes for all alpha spectrometry sample and background counts.

D=decay correction for time "t" (in days) from sample collection to sample counting, given by: $D=e^{-0.0004t}$

5.2 Procedure for Calculating Emission Rate in Curies per Metric Ton of Phosphate Rock Processed. Calculate the polonium-210 emission per metric ton of rock processed from each run at each stack using Equation 111-4. The emission rate from each stack is determined by averaging the emission rates calculated for each of the three runs at each stack.

$$R_s = \frac{1 \times 10^{-12} A Q_{210}}{V_{210} M_H} \quad \text{Eq. 111-4}$$

Where:
 R_s =emission rate from stack, in curies of polonium-210 per metric ton of rock processed.
 A =picocuries of polonium-210 in the particulate sample as determined by A in Eq. 111-3.
 Q_{210} =volumetric flow rate of effluent stream in dry standard m³/hr as determined by Method 2 of Appendix A to 40 CFR Part 60.
 V_{210} =total volume of air sample in dry standard m³ as determined by Method 5 of Appendix A to 40 CFR Part 60.
 M_H =rock processing rate during sampling in metric tons/hr.
 1×10^{-12} =curies per picocurie.

5.3 Average Stack Emission Rate Calculation. Determine the average stack emission rate from the average of the three emission rates calculated in Section 5.2. Perform these calculations for each stack of each calciner.

5.4 Calciner Emission Rate Calculation. Determine each calciner's emission rate (X_i) by taking the sum of the emission rates from all stacks of each calciner.

5.5 Annual Polonium-210 Emission Calculation. Determine the annual elemental phosphorus plant emissions of polonium-210 by taking the sum of emission rates at each calciner (X_i in 5.4) and multiplying this sum by the annual metric tons of phosphate rock processed by that calciner, according to Eq. 111-5.

$$S = X_1 M_1 + X_2 M_2 + \dots + X_n M_n \quad \text{Eq. 111-5}$$

Where:
 S =annual polonium-210 emissions in curies from the elemental phosphorus plant.
 X_i =emission rate from a calciner (i) in curies per metric ton, as determined in Section 5.4.

N =number of calciners at the elemental phosphorus plant.
 M_i =phosphate rock processed per year, in metric tons for each calciner.

6.0 Standardization of Alpha Spectrometry System

6.1 Obtain a standardized solution of an alpha-emitting actinide element such as plutonium-239 or americium-241. Add a quantity of the standardized solution to a 100 ml volumetric flask so that the final concentration when diluted to a volume of 100 ml will be approximately 1 pCi/ml. Add 10 ml of 16 M HNO₃ and dilute to 100 ml with distilled water.

6.2 Add 20 ml of 1 M HCl to each of six 150 ml beakers.

6.3 Add 1.0 ml of lanthanum carrier, 0.1 mg lanthanum per ml, to the acid solution in each beaker.

6.4 Add 1.0 ml of actinide solution from Section 6.1 to each beaker.

6.5 Add 5.0 ml of 3 M HF to each beaker.

6.6 Cover beakers and allow solutions to stand for a minimum of 30 minutes.

6.7 Filter each solution through a filter membrane using this suction filter apparatus.

6.8 After each filtration, wash the filter membrane with 10 ml of distilled water and 5 ml of ethanol.

6.9 Allow the filter membrane to air dry on the filter apparatus.

6.10 Carefully remove the filter membrane and mount with double-side tape on the inner surface of a planchet. Mount filter with filtration side up.

6.11 Place planchet in an alpha spectrometry system and count each planchet for 1000 minutes.

6.12 The counting efficiency of each detector can be calculated using Eq. 111-6.

$$E_c = \frac{C_s - C_b}{2.22 A_s T} \quad \text{Eq. 111-6}$$

where:
 C_s =gross counts in actinide peak.
 C_b =background counts in same peak area as C_s .
 2.22 =disintegrations per minute per picocurie.
 A_s =picocuries of actinide added.
 E_c =counting efficiency, counts per minute per disintegration per minute.
 T =counting time in minutes, specified in Section 8.11 as 1000 minutes.

6.13 Determine the average counting efficiency for each detector by calculating the average of the six determinations.

7.0 Preparation of Standardized Solution of Polonium-209

7.1 Obtain polonium-209 solution from an available supplier. Add a quantity of the Po-209 solution to a 100 ml volumetric flask so that the final concentration when diluted to a 100 ml volume will be approximately 1 pCi/ml. Add 10 ml of 16 M HNO₃ and dilute to 100 ml with distilled water.

7.2 Add 20 ml of 1 M HCl to each of six 150 ml beakers.

7.3 Add 1.0 ml of lanthanum carrier, 0.1 mg lanthanum per ml, to the acid solution in each beaker.

7.4 Add 1.0 ml of polonium-209 tracer from Section 7.1 to each beaker.

7.5 Add 3.0 ml of 15 M ammonium hydroxide to each beaker.

7.6 Cover beakers and allow to stand for a minimum of 30 minutes.

7.7 Filter the contents of each beaker through a separate filter membrane.

7.8 After each filtration, wash membrane with 10 ml of distilled water and 5 ml of ethanol.

7.9 Allow filter membrane to dry on filter apparatus.

7.10 Carefully remove the filter membrane and mount with double-side tape on the inner surface of a planchet. Mount filter with filtration side up.

7.11 Place planchet in alpha spectrometry system and count each planchet for 1000 minutes.

7.12 The activity of the polonium solution can be calculated using Eq. 111-7.

$$F = \frac{C_s - C_b}{2.22 E_c T} \quad \text{Eq. 111-7}$$

where:

F=activity of polonium-209 solution, in pCi.
C_s=gross counts of polonium-209 in the 4.88 MeV region of the spectrum in the counting time T.

C_b=background counts in the 4.88 MeV region of spectrum the in the counting time T.
2.22=disintegrations per minute per picocurie.

E_c=counting efficiency of detector used, counts per minute per disintegration per minute.

T=counting time, specified in Section 7.11 as 1000 minutes.

7.13 Determine the average activity of the polonium-209 solution from the six determinations.

7.14 Aliquots of the solution from Section 7.1 are to be used as tracer with each polonium-210 analysis.

8.0 Standardization of Internal Proportional Counter

8.1 Obtain a standardized solution of alpha-emitting actinide element such as polonium-239 or americium-241. Add a quantity of the standardized solution to a 100 ml volumetric flask so that the final concentration when diluted to a 100 ml volume will be approximately 100 pCi/ml. Add 10 ml of 16 M HNO₃ and dilute to 100 ml with distilled water.

8.2 Add 20 ml of 1 M HCl to each of six 150 ml beakers.

8.3 Add 1.0 ml of lanthanum carrier, 0.1 mg lanthanum per ml, to the acid solution in each beaker.

8.4 Add 1.0 ml of the actinide solution from Section 8.1 to each beaker.

8.5 Add 5.0 ml of 3 M HF to each beaker.

8.6 Cover beakers and allow solutions to stand for a minimum of 30 minutes.

8.7 Filter each solution through a filter membrane using the suction filter apparatus.

8.8 After each filtration, wash membrane with 10 ml of distilled water and 5 ml of ethanol.

8.9 Allow filter membrane to dry on filter apparatus.

8.10 Carefully remove filter membrane and mount with double-side tape on the inner surface of a planchet. Mount filter with filtration side up.

8.11 Place planchet in internal proportional counter and count for 100 minutes.

8.12 The counting efficiency of the internal proportional counter is determined as follows from the six samples:

$$E_s = \frac{C_s - C_b}{2.22 A_A T} \quad \text{Eq. 111-8}$$

where:

E_s=counting efficiency of proportional counter, counts per minute per disintegration per minute.

C_s=gross counts of standard.

C_b=gross counts of procedure background.
2.22=disintegrations per minute per picocurie.

A_A=picocuries of actinide added.

T=counting time in minutes, specified in Section 8.11 as 100 minutes.

8.13 Determine the average counting efficiency of the six determinations.

9.0 Quality Assurance

9.1 General Requirements.

9.1.1 All analysts using this method are required to demonstrate their ability to

the method and to define their respective accuracy and precision criteria.

9.2 Specific Requirements.

9.2.1 Each sample will be analyzed in duplicate.

9.2.2 Every tenth sample will be an externally prepared performance evaluation sample submitted by the Quality Assurance Officer.

9.2.3 Duplicate measurements are considered acceptable when the difference between them is less than two standard deviations as described in EPA 600/4-77-001 or subsequent revisions.

METHOD 114—TEST METHODS FOR MEASURING RADIONUCLIDE EMISSIONS FROM STATIONARY SOURCES

1. Purpose and Background

This method provides the requirements for: (1) Stack monitoring and sample collection methods appropriate for radionuclides; (2) radiochemical methods which are used in determining the amounts of radionuclides collected by the stack sampling and; (3) quality assurance methods which are conducted in conjunction with these measurements. These methods are appropriate for emissions for stationary sources. A list of references is provided.

Many different types of facilities release radionuclides into air. These radionuclides differ in the chemical and physical forms, half-lives and type of radiation emitted. The appropriate combination of sample extraction, collection and analysis for an individual radionuclide is dependent upon many interrelated factors including the mixture of other radionuclides present. Because of this wide range of conditions, no single method for monitoring or sample collection and analysis of a radionuclide is applicable to all types of facilities. Therefore, a series of methods based on "principles of measurement" are described for monitoring and sample collection and analysis which are applicable to the measurement of radionuclides found in effluent streams at stationary sources. This approach provides the user with the flexibility to choose the most appropriate combination of monitoring and sample collection and analysis methods which are applicable to the effluent stream to be measured.

2. Stack Monitoring and Sample Collection Methods

Monitoring and sample collection methods are described based on "principles of monitoring and sample collection" which are applicable to the measurement of radionuclides from effluent streams at stationary sources. Radionuclides of most elements will be in the particulate form in these effluent streams and can be readily collected using a suitable filter media. Radionuclides of hy-

drogen, oxygen, carbon, nitrogen, the noble gases and in some circumstances iodine will be in the gaseous form. Radionuclides of these elements will require either the use of an in-line or off-line monitor to directly measure the radionuclides, or suitable sorbers, condensers or bubblers to collect the radionuclides.

2.1 Radionuclides as Particulates. The extracted effluent stream is passed through a filter media to remove the particulates. The filter must have a high efficiency for removal of sub-micron particles. The guidance in ANSI N13.1—1969 shall be followed in using filter media to collect particulates (incorporated by reference—see § 61.18).

2.2 Radionuclides as Gases.

2.2.1 The Radionuclide Tritium (H-3). Tritium in the form of water vapor is collected from the extracted effluent sample by sorption, condensation or dissolution techniques. Appropriate collectors may include silica gel, molecular sieves, and ethylene glycol or water bubblers.

Tritium in the gaseous form may be measured directly in the sample stream using Method B-1, collected as a gas sample or may be oxidized using a metal catalyst to tritiated water and collected as described above.

2.2.2 Radionuclides of Iodine. Iodine is collected from an extracted sample by sorption or dissolution techniques. Appropriate collectors may include charcoal, impregnated charcoal, metal zeolite and caustic solutions.

2.2.3 Radionuclides of Argon, Krypton and Xenon. Radionuclides of these elements are either measured directly by an in-line or off-line monitor, or are collected from the extracted sample by low temperature sorption techniques. Appropriate sorbers may include charcoal or metal zeolite.

2.2.4 Radionuclides of Oxygen, Carbon, Nitrogen and Radon. Radionuclides of these elements are measured directly using an in-line or off-line monitor. Radionuclides of carbon in the form of carbon dioxide may be collected by dissolution in caustic solutions.

2.3 Definition of Terms

In-line monitor means a continuous measurement system in which the detector is placed directly in or adjacent to the effluent stream. This may involve either gross radioactivity measurements or specific radionuclide measurements. Gross measurements shall be made in conformance with the conditions specified in Methods A-4, B-2 and G-4.

Off-line monitor means a measurement system in which the detector is used to continuously measure an extracted sample of the effluent stream. This may involve either gross radioactivity measurements or specific radionuclide measurements. Gross measure-

ments shall be made in conformance with the conditions specified in Methods A-4, B-2 and G-4.

Sample collection means a procedure in which the radionuclides are removed from an extracted sample of the effluent using a collection media. These collection media include filters, absorbers, bubblers and condensers. The collected sample is analyzed using the methods described in Section 3.

3. Radionuclide Analysis Methods

A series of methods based on "principles of measurement" are described which are applicable to the analysis of radionuclides collected from airborne effluent streams at stationary sources. These methods are applicable only under the conditions stated and within the limitations described. Some methods specify that only a single radionuclide be present in the sample or the chemically separated sample. This condition should be interpreted to mean that no other radionuclides are present in quantities which would interfere with the measurement.

Also identified (Table 1) are methods for a selected list of radionuclides. The listed radionuclides are those which are most commonly used and which have the greatest potential for causing dose to members of the public. Use of methods based on principles of measurement other than those described in this section must be approved in advance of use by the Administrator. For radionuclides not listed in Table 1, any of the described methods may be used provided the user can demonstrate that the applicability conditions of the method have been met.

The type of method applicable to the analysis of a radionuclide is dependent upon the type of radiation emitted, i.e., alpha, beta or gamma. Therefore, the methods described below are grouped according to principles of measurements for the analysis of alpha, beta and gamma emitting radionuclides.

3.1 Methods for Alpha Emitting Radionuclides

3.1.1 Method A-1, Radiochemistry-Alpha Spectrometry.

Principle: The element of interest is separated from other elements, and from the sample matrix using radiochemical techniques. The procedure may involve precipitation, ion exchange, or solvent extraction. Carriers (elements chemically similar to the element of interest) may be used. The element is deposited on a planchet in a very thin film by electrodeposition or by coprecipitation on a very small amount of carrier, such as lanthanum fluoride. The deposited element is then counted with an alpha spectrometer. The activity of the nuclide of interest is measured by the number of alpha counts in the appropriate energy region. A correction for chemical yield and counting

efficiency is made using a standardized radioactive nuclide (tracer) of the same element. If a radioactive tracer is not available for the element of interest, a predetermined chemical yield factor may be used.

Applicability: This method is applicable for determining the activity of any alpha-emitting radionuclide, regardless of what other radionuclides are present in the sample provided the chemical separation step produces a very thin sample and removes all other radionuclides which could interfere in the spectral region of interest. APHA-605(2), ASTM-D-3972(13).

3.1.2 Method A-2, Radiochemistry-Alpha Counting.

Principle: The element of interest is separated from other elements, and from the sample matrix using radiochemistry. The procedure may involve precipitation, ion exchange, or solvent extraction. Carriers (elements chemically similar to the element of interest) may be used. The element is deposited on a planchet in a thin film and counted with an alpha counter. A correction for chemical yield (if necessary) is made. The alpha count rate measures the total activity of all emitting radionuclides of the separated element.

Applicability: This method is applicable for the measurement of any alpha-emitting radionuclide, provided no other alpha emitting radionuclide is present in the separated sample. It may also be applicable for determining compliance, when other radionuclides of the separated element are present, provided that the calculated emission rate is assigned to the radionuclide which could be present in the sample that has the highest dose conversion factor. IDO-12096(18).

3.1.3 Method A-3, Direct Alpha Spectrometry.

Principle: The sample, collected on a suitable filter, is counted directly on an alpha spectrometer. The sample must be thin enough and collected on the surface of the filter so that any absorption of alpha particle energy in the sample or the filter, which would degrade the spectrum, is minimal.

Applicability: This method is applicable to simple mixtures of alpha emitting radionuclides and only when the amount of particulates collected on the filter paper are relatively small and the alpha spectra is adequately resolved. Resolutions should be 50 keV (FWHM) or better, ASTM-D-3084(16).

3.1.4 Method A-4, Direct Alpha Counting (Gross alpha determination).

Principle: The sample, collected on a suitable filter, is counted with an alpha counter. The sample must be thin enough so that self-absorption is not significant and the filter must be of such a nature that the particles are retained on the surface.

Applicability: Gross alpha determinations may be used to measure emissions of speci-

ic radionuclides only (1) when it is known that the sample contains only a single radionuclide, or the identity and isotopic ratio of the radionuclides in the sample are well-known, and (2) measurements using either Method A-1, A-2 or A-5 have shown that this method provides a reasonably accurate measurement of the emission rate. Gross alpha measurements are applicable to unidentified mixtures of radionuclides only for the purposes and under the conditions described in section 3.7. APHA-601(3), ASTM-D-1943(10).

3.1.5 Method A-5, Chemical Determination of Uranium.

Principle: Uranium may be measured chemically by either colorimetry or fluorometry. In both procedures, the sample is dissolved, the uranium is oxidized to the hexavalent form and extracted into a suitable solvent. Impurities are removed from the solvent layer. For colorimetry, dibenzoylmethane is added, and the uranium is measured by the absorbance in a colorimeter. For fluorometry, a portion of the solution is fused with a sodium fluoride-lithium fluoride flux and the uranium is determined by the ultraviolet activated fluorescence of the fused disk in a fluorometer.

Applicability: This method is applicable to the measurements of emission rates of uranium when the isotopic ratio of the uranium radionuclides is well known. ASTM-E-318(15), ASTM-D-2907(14).

3.1.6 Method A-6, Radon-222—Continuous Gas Monitor.

Principle: Radon-222 is measured directly in a continuously extracted sample stream by passing the air stream through a calibrated scintillation cell. Prior to the scintillation cell, the air stream is treated to remove particulates and excess moisture. The alpha particles from radon-222 and its decay products strike a zinc sulfide coating on the inside of the scintillation cell producing light pulses. The light pulses are detected by a photomultiplier tube which generates electrical pulses. These pulses are processed by the system electronics and the read out is in pCi/l of radon-222.

Applicability: This method is applicable to the measurement of radon-222 in effluent streams which do not contain significant quantities of radon-220. Users of this method should calibrate the monitor in a radon calibration chamber at least twice per year. The background of the monitor should also be checked periodically by operating the instrument in a low radon environment. EPA 520/1-89-009(24).

3.1.7 Method A-7, Radon-222-Alpha Track Detectors

Principle: Radon-222 is measured directly in the effluent stream using alpha track detectors (ATD). The alpha particles emitted by radon-222 and its decay products strike a small plastic strip and produce submicron

damage tracks. The plastic strip is placed in a caustic solution that accentuates the damage tracks which are counted using a microscope or automatic counting system. The number of tracks per unit area is correlated to the radon concentration in air using a conversion factor derived from data generated in a radon calibration facility.

Applicability: Prior approval from EPA is required for use of this method. This method is only applicable to effluent streams which do not contain significant quantities of radon-220, unless special detectors are used to discriminate against radon-220. This method may be used only when ATDs have been demonstrated to produce data comparable to data obtained with Method A-6. Such data should be submitted to EPA when requesting approval for the use of this method. EPA 520/1-89-009(24).

3.2 Methods for Gaseous Beta Emitting Radionuclides.

3.2.1 Method B-1, Direct Counting in Flow-Through Ionization Chambers.

Principle: An ionization chamber containing a specific volume of gas which flows at a given flow rate through the chamber is used. The sample (effluent stream sample) acts as the counting gas for the chamber. The activity of the radionuclide is determined from the current measured in the ionization chamber.

Applicability: This method is applicable for measuring the activity of a gaseous beta-emitting radionuclide in an effluent stream that is suitable as a counting gas, when no other beta-emitting nuclides are present. DOE/EP-0096(17), NCRP-58(23).

3.2.2 Method B-2, Direct Counting With In-line or Off-line Beta Detectors.

Principle: The beta detector is placed directly in the effluent stream (in-line) or an extracted sample of the effluent stream is passed through a chamber containing a beta detector (off-line). The activities of the radionuclides present in the effluent stream are determined from the beta count rate, and a knowledge of the radionuclides present and the relationship of the gross beta count rate and the specific radionuclide concentration.

Applicability: This method is applicable only to radionuclides with maximum beta particle energies greater than 0.2 MeV. This method may be used to measure emissions of specific radionuclides only when it is known that the sample contains only a single radionuclide or the identity and isotopic ratio of the radionuclides in the effluent stream are well known. Specific radionuclide analysis of periodic grab samples may be used to identify the types and quantities of radionuclides present and to establish the relationship between specific radionuclide analyses and gross beta count rates.

This method is applicable to unidentified mixtures of gaseous radionuclides only for the purposes and under the conditions described in section 3.7.

3.3 Methods for Non-Gaseous Beta Emitting Radionuclides.

3.3.1 Method B-3, Radiochemistry-Beta Counting.

Principle: The element of interest is separated from other elements, and from the sample matrix by radiochemistry. This may involve precipitation, distillation, ion exchange, or solvent extraction. Carriers (elements chemically similar to the element of interest) may be used. The element is deposited on a planchet, and counted with a beta counter. Corrections for chemical yield, and decay (if necessary) are made. The beta count rate determines the total activity of all radionuclides of the separated element. This method may also involve the radiochemical separation and counting of a daughter element, after a suitable period of ingrowth, in which case it is specific for the parent nuclide.

Applicability: This method is applicable for measuring the activity of any beta-emitting radionuclide, with a maximum energy greater than 0.2 MeV, provided no other radionuclide is present in the separated sample. APHA-608(5).

3.3.2 Method B-4, Direct Beta Counting (Gross beta determination).

Principle: The sample, collected on a suitable filter, is counted with a beta counter. The sample must be thin enough so that self-absorption corrections can be made.

Applicability: Gross beta measurements are applicable only to radionuclides with maximum beta particle energies greater than 0.2 MeV. Gross beta measurements may be used to measure emissions of specific radionuclides only (1) when it is known that the sample contains only a single radionuclide, and (2) measurements made using Method B-3 show reasonable agreement with the gross beta measurement. Gross beta measurements are applicable to mixtures of radionuclides only for the purposes and under the conditions described in section 3.7. APHA-602(4), ASTM-D-1890(11).

3.3.3 Method B-5, Liquid Scintillation Spectrometry.

Principle: An aliquot of a collected sample or the result of some other chemical separation or processing technique is added to a liquid scintillation "cocktail" which is viewed by photomultiplier tubes in a liquid scintillation spectrometer. The spectrometer is adjusted to establish a channel or "window" for the pulse energy appropriate to the nuclide of interest. The activity of the nuclide of interest is measured by the counting rate in the appropriate energy channel. Corrections are made for chemical yield where separations are made.

Applicability: This method is applicable to any beta-emitting nuclide when no other radionuclide is present in the sample or the separated sample provided that it can be incorporated in the scintillation cocktail. This method is also applicable for samples which contain more than one radionuclide but only when the energies of the beta particles are sufficiently separated so that they can be resolved by the spectrometer. This method is most applicable to the measurement of low-energy beta emitters such as tritium and carbon-14. APHA-609(6), EMLV-539-17(19).

3.4 Gamma Emitting Radionuclides

3.4.1 Method G-1, High Resolution Gamma Spectrometry.

Principle: The sample is counted with a high resolution gamma detector, usually either a Ge(Li) or a high purity Ge detector, connected to a multichannel analyzer or computer. The gamma emitting radionuclides in the sample are measured from the gamma count rates in the energy regions characteristic of the individual radionuclide. Corrections are made for counts contributed by other radionuclides to the spectral regions of the radionuclides of interest. Radiochemical separations may be made prior to counting but are usually not necessary.

Applicability: This method is applicable to the measurement of any gamma emitting radionuclide with gamma energies greater than 20 keV. It can be applied to complex mixtures of radionuclides. The samples counted may be in the form of particulate filters, absorbers, liquids or gases. The method may also be applied to the analysis of gaseous gamma emitting radionuclides directly in an effluent stream by passing the stream through a chamber or cell containing the detector. ASTM-3649(9), IDO-12096(18).

3.4.2 Method G-2, Low Resolution Gamma Spectrometry.

Principle: The sample is counted with a low resolution gamma detector, a thallium activated sodium iodide crystal. The detector is coupled to a photomultiplier tube and connected to a multichannel analyzer. The gamma emitting radionuclides in the sample are measured from the gamma count rates in the energy regions characteristic of the individual radionuclides. Corrections are made for counts contributed by other radionuclides to the spectral regions of the radionuclides of interest. Radiochemical separation may be used prior to counting to obtain less complex gamma spectra if needed.

Applicability: This method is applicable to the measurement of gamma emitting radionuclides with energies greater than 100 keV. It can be applied only to relatively simple mixtures of gamma emitting radionuclides. The samples counted may be in the

form of particulate filters, absorbers, liquids or gas. The method can be applied to the analysis of gaseous radionuclides directly in an effluent stream by passing the gas stream through a chamber or cell containing the detector. ASTM-D-2459(12), EMSL-LV-0539-17(19).

3.4.3 Method G-3, Single Channel Gamma Spectrometry.

Principle: The sample is counted with a thallium activated sodium iodide crystal. The detector is coupled to a photomultiplier tube connected to a single channel analyzer. The activity of a gamma emitting radionuclide is determined from the gamma counts in the energy range for which the counter is set.

Applicability: This method is applicable to the measurement of a single gamma emitting radionuclide. It is not applicable to mixtures of radionuclides. The samples counted may be in the form of particulate filters, absorbers, liquids or gas. The method can be applied to the analysis of gaseous radionuclides directly in an effluent stream by passing the gas stream through a chamber or cell containing the detector.

3.4.4 Method G-4, Gross Gamma Counting.

Principle: The sample is counted with a gamma detector usually a thallium activated sodium iodine crystal. The detector is coupled to a photomultiplier tube and gamma rays above a specific threshold energy level are counted.

Applicability: Gross gamma measurements may be used to measure emissions of specific radionuclides only when it is known that the sample contains a single radionuclide or the identity and isotopic ratio of the radionuclides in the effluent stream are well known. When gross gamma measurements are used to determine emissions of specific radionuclides periodic measurements using Methods G-1 or G-2 should be made to demonstrate that the gross gamma measurements provide reliable emission data. This method may be applied to analysis of gaseous radionuclides directly in an effluent stream by placing the detector directly in or adjacent to the effluent stream or passing an extracted sample of the effluent stream through a chamber or cell containing the detector.

3.5 Counting Methods. All of the above methods with the exception of Method A-5 involve counting the radiation emitted by the radionuclide. Counting methods applicable to the measurement of alpha, beta and gamma radiations are listed below. The equipment needed and the counting principles involved are described in detail in ASTM-3648(8).

3.5.1 Alpha Counting:

• **Gas Flow Proportional Counters.** The alpha particles cause ionization in the counting gas and the resulting electrical

pulses are counted. These counters may be windowless or have very thin windows.

• **Scintillation Counters.** The alpha particles transfer energy to a scintillator resulting in a production of light photons which strike a photomultiplier tube converting the light photons to electrical pulses which are counted. The counters may involve the use of solid scintillation materials such as zinc sulfide or liquid scintillation solutions.

• **Solid-State Counters.** Semiconductor materials, such as silicon surface-barrier p-n junctions, act as solid ionization chambers. The alpha particles interact which the detector producing electron hole pairs. The charged pair is collected by an applied electrical field and the resulting electrical pulses are counted.

• **Alpha Spectrometers.** Semiconductor detectors used in conjunction with multichannel analyzers for energy discrimination.

3.5.2 Beta Counting:

• **Ionization Chambers.** These chambers contain the beta-emitting nuclide in gaseous form. The ionization current produced is measured.

• **Geiger-Muller (GM) Counters or Gas Flow Proportional Counters.** The beta particles cause ionization in the counting gas and the resulting electrical pulses are counted. Proportional gas flow counters which are heavily shielded by lead or other metal, and provided with an anti-coincidence shield to reject cosmic rays, are called low background beta counters.

• **Scintillation Counters.** The beta particles transfer energy to a scintillator resulting in a production of light photons, which strike a photomultiplier tube converting the light photon to electrical pulses which are counted. This may involve the use of anthracene crystals, plastic scintillator, or liquid scintillation solutions with organic phosphors.

• **Liquid Scintillation Spectrometers.** Liquid scintillation counters which use two photomultiplier tubes in coincidence to reduce background counts. This counter may also electronically discriminate among pulses of a given range of energy.

3.5.3 Gamma Counting:

• **Low-Resolution Gamma Spectrometers.** The gamma rays interact with thallium activated sodium iodide or cesium iodide crystal resulting in the release of light photons which strike a photomultiplier tube converting the light pulses to electrical pulses proportional to the energy of the gamma ray. Multi-channel analyzers are used to separate and store the pulses according to the energy absorbed in the crystal.

• **High-Resolution gamma Spectrometers.** Gamma rays interact with a lithium-drifted (Ge(Li)) or high-purity germanium (HPGe) semiconductor detectors resulting in a production of electron-hole pairs. The charged

pair is collected by an applied electrical field. A very stable low noise preamplifier amplifies the pulses of electrical charge resulting from the gamma photon interactions. Multichannel analyzers or computers are used to separate and store the pulses according to the energy absorbed in the crystal.

• *Single Channel Analyzers.* Thallium activated sodium iodide crystals used with a single window analyzer. Pulses from the photomultiplier tubes are separated in a single predetermined energy range.

3.5.4 Calibration of Counters. Counters are calibrated for specific radionuclide measurements using a standard of the radionuclide under either identical or very similar conditions as the sample to be counted. For gamma spectrometers a series of standards covering the energy range of interest may be used to construct a calibration curve relating gamma energy to counting efficiency.

In those cases where a standard is not available for a radionuclide, counters may be calibrated using a standard with energy characteristics as similar as possible to the radionuclide to be measured. For gross alpha and beta measurements of the unidentified mixtures of radionuclides, alpha counters are calibrated with a natural uranium standard and beta counters with a cesium-137 standard. The standard must contain the same weight and distribution of solids as the samples, and be mounted in an identical manner. If the samples contain variable amounts of solids, calibration curves relating weight of solids present to counting efficiency are prepared. Standards other than those prescribed may be used provided it can be shown that such standards are more applicable to the radionuclide mixture measured.

3.6 Radiochemical Methods for Selected Radionuclides. Methods for a selected list of radionuclides are listed in Table 1. The radionuclides listed are those which are most commonly used and which have the greatest potential for causing doses to members of the public. For radionuclides not listed in Table 1, methods based on any of the applicable "principles of measurement" described in section 3.1 through 3.4 may be used.

3.7 Applicability of Gross Alpha and Beta Measurements to Unidentified Mixtures of Radionuclides. Gross alpha and beta measurements may be used as a screening measurement as a part of an emission measurement program to identify the need to do specific radionuclide analyses or to confirm or verify that unexpected radionuclides are not being released in significant quantities.

Gross alpha (Method A-4) or gross beta (Methods B-2 or B-4) measurements may also be used for the purpose of comparing the measured concentrations in the effluent

stream with the limiting "Concentration Levels for Environmental Compliance" in Table 2 of Appendix E. For unidentified mixtures, the measured concentration value shall be compared with the lowest environmental concentration limit for any radionuclide which is not known to be absent from the effluent stream.

TABLE 1—LIST OF APPROVED METHODS FOR SPECIFIC RADIONUCLIDES

Radionuclide	Approved methods of analysis
Am-241	A-1, A-2, A-3, A-4
Ar-41	B-1, B-2, G-1, G-2, G-3, G-4
Ba-140	G-1, G-2, G-3, G-4
Br-82	G-1, G-2, G-3, G-4
C-11	B-1, B-2, B-3, G-1, G-2, G-3, G-4
C-14	B-5
Ca-45	B-3, B-4, B-5
Ce-144	G-1, G-2, G-3, G-4
Cm-244	A-1, A-2, A-3, A-4
Co-60	G-1, G-2, G-3, G-4
Cr-51	G-1, G-2, G-3, G-4
Cs-134	G-1, G-2, G-3, G-4
Cs-137	G-1, G-2, G-3, G-4
Fe-55	B-5, G-1
Fe-59	G-1, G-2, G-3, G-4
Ga-67	G-1, G-2, G-3, G-4
H-3 (H ₂ O)	B-5
H-3 (gas)	B-1
I-123	G-1, G-2, G-3, G-4
I-125	G-1
I-131	G-1, G-2, G-3, G-4
In-113m	G-1, G-2, G-3, G-4
Ir-192	G-1, G-2, G-3, G-4
Kr-85	B-1, B-2, B-3, G-1, G-2, G-3, G-4
Kr-87	B-1, B-2, G-1, G-2, G-3, G-4
Kr-88	B-1, B-2, G-1, G-2, G-3, G-4
Mn-54	G-1, G-2, G-3, G-4
Mo-99	G-1, G-2, G-3, G-4
N-13	B-1, B-2, G-1, G-2, G-3, G-4
O-15	B-1, B-2, G-1, G-2, G-3, G-4
P-32	B-3, B-4, B-5
Pm-147	B-3, B-4, B-5
Po-210	A-1, A-2, A-3, A-4
Pu-238	A-1, A-2, A-3, A-4
Pu-239	A-1, A-2, A-3, A-4
Pu-240	A-1, A-2, A-3, A-4
S-35	B-5
Se-75	G-1, G-2, G-3, G-4
Sr-90	B-3, B-4, B-5
Tc-99	B-3, B-4, B-5
Te-201	G-1, G-2, G-3, G-4
Uranium (total alpha)	A-1, A-2, A-3, A-4
Uranium (isotopic)	A-1, A-3
Uranium (Natural)	A-5
Xe-133	G-1
Yb-169	G-1, G-2, G-3, G-4
Zn-65	G-1, G-2, G-3, G-4

4. Quality Assurance Methods

Each facility required to measure their radionuclide emissions shall conduct a quality assurance program in conjunction with the radionuclide emission measurements. This program shall assure that the emission measurements are representative, and are of

known precision and accuracy and shall include administrative controls to assure prompt response when emission measurements indicate unexpectedly large emissions. The program shall consist of a system of policies, organizational responsibilities, written procedures, data quality specifications, audits, corrective actions and reports. This quality assurance program shall include the following program elements:

4.1 The organizational structure, functional responsibilities, levels of authority and lines of communications for all activities related to the emissions measurement program shall be identified and documented.

4.2 Administrative controls shall be prescribed to ensure prompt response in the event that emission levels increase due to unplanned operations.

4.3 The sample collection and analysis procedures used in measuring the emissions shall be described including where applicable:

4.3.1 Identification of sampling sites and number of sampling points, including the rationale for site selections.

4.3.2 A description of sampling probes and representativeness of the samples.

4.3.3 A description of any continuous monitoring system used to measure emissions, including the sensitivity of the system, calibration procedures and frequency of calibration.

4.3.4 A description of the sample collection systems for each radionuclide measured, including frequency of collection, calibration procedures and frequency of calibration.

4.3.5 A description of the laboratory analysis procedures used for each radionuclide measured, including frequency of analysis, calibration procedures and frequency of calibration.

4.3.6 A description of the sample flow rate measurement systems or procedures, including calibration procedures and frequency of calibration.

4.3.7 A description of the effluent flow rate measurement procedures, including frequency of measurements, calibration procedures and frequency of calibration.

4.4 The objectives of the quality assurance program shall be documented and shall state the required precision, accuracy and completeness of the emission measurement data including a description of the procedures used to assess these parameters. Accuracy is the degree of agreement of a measurement with a true or known value. Precision is a measure of the agreement among individual measurements of the same parameters under similar conditions. Completeness is a measure of the amount of valid data obtained compared to the amount expected under normal conditions.

4.5 A quality control program shall be established to evaluate and track the quality of the emissions measurement data against preset criteria. The program should include where applicable a system of replicates, spiked samples, split samples, blanks and control charts. The number and frequency of such quality control checks shall be identified.

4.6 A sample tracking system shall be established to provide for positive identification of samples and data through all phases of the sample collection, analysis and reporting system. Sample handling and preservation procedures shall be established to maintain the integrity of samples during collection, storage and analysis.

4.7 Periodic internal and external audits shall be performed to monitor compliance with the quality assurance program. These audits shall be performed in accordance with written procedures and conducted by personnel who do not have responsibility for performing any of the operations being audited.

4.8 A corrective action program shall be established including criteria for when corrective action is needed, what corrective actions will be taken and who is responsible for taking the corrective action.

4.9 Periodic reports to responsible management shall be prepared on the performance of the emissions measurements program. These reports should include assessment of the quality of the data, results of audits and description of corrective actions.

4.10 The quality assurance program should be documented in a quality assurance project plan which should address each of the above requirements.

5. References

(1) American National Standards Institute, "Guide to Sampling Airborne Radioactive Materials in Nuclear Facilities", ANSI-N13.1-1969, American National Standards Institute, New York, New York (1969).

(2) American Public Health Association, "Methods of Air Sampling", 2nd Edition, Method 605, "Tentative Method of Analysis for Plutonium Content of Atmospheric Particulate Matter", American Public Health Association, New York, NY (1977).

(3) Ibid, Method 601, "Tentative Method of Analysis for Gross Alpha Radioactivity Content of the Atmosphere".

(4) Ibid, Method 602, "Tentative Method of the Analysis for Gross Beta Radioactivity Content of the Atmosphere".

(5) Ibid, Method 608, "Tentative Method of Analysis for Strontium-90 Content of Atmospheric Particulate Matter".

(6) Ibid, Method 609, "Tentative Method of Analysis for Tritium Content of the Atmosphere".

(7) Ibid, Method 603, "Tentative Method of Analysis for Iodine-131 Content of the Atmosphere".

(8) American Society for Testing and Materials, 1986 Annual Book ASTM Standards, Designation D-3648-78, "Standard Practices for the Measurement of Radioactivity", American Society for Testing and Materials, Philadelphia, PA (1986).

(9) Ibid, Designation D-3649-85, "Standard Practice for High Resolution Gamma Spectrometry".

(10) Ibid, Designation D-1943-81, "Standard Test Method for Alpha Particle Radioactivity of Water".

(11) Ibid, Designation D-1890-81, "Standard Test Method for Beta Particle Radioactivity of Water".

(12) Ibid, Designation D-2459-72, "Standard Test Method for Gamma Spectrometry of Water".

(13) Ibid, Designation D-3972-82, "Standard Test Method for Isotopic Uranium in Water by Radiochemistry".

(14) Ibid, Designation D-2907-83, "Standard Test Methods for Microquantities of Uranium in Water by Fluorometry".

(15) Ibid, Designation E-318, "Standard Test Method for Uranium in Aqueous Solutions by Colorimetry".

(16) Ibid, Designation D-3084-75, "Standard Practice for Alpha Spectrometry of Water".

(17) Corley, J.P. and C.D. Corbit, "A Guide for Effluent Radiological Measurements at DOE Installations", DOE/EP-0096, Pacific Northwest Laboratories, Richland, Washington (1983).

(18) Department of Energy, "RESL Analytical Chemistry Branch Procedures Manual", IDO-12096, U.S. Department of Energy, Idaho Falls, Idaho (1982).

(19) Environmental Protection Agency, "Radiochemical Analytical Procedures for Analysis of Environmental Samples", EMSL-LV-0539-17, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Las Vegas, Nevada (1979).

(20) Environmental Protection Agency, "Radiochemistry Procedures Manual", EPA 520/5-84-006, Eastern Environmental Radiation Facility, Montgomery, Alabama (1984).

(21) National Council on Radiation Protection and Measurements, NCRP Report No. 50, "Environmental Radiation Measurements", National Council on Radiation Protection and Measurement, Bethesda, Maryland (1976).

(22) Ibid, Report No. 47, "Tritium Measurement Techniques", (1976).

(23) Ibid, Report No. 58 "A Handbook of Radioactivity Measurement Procedures" (1985).

(24) Environmental Protection Agency, "Indoor Radon and Radon Decay Product

Measurement Protocols", EPA 520/1-89-009, U.S. Environmental Protection Agency, Washington, DC (1989).

METHOD 115—MONITORING FOR RADON-222 EMISSIONS

This Appendix describes the monitoring methods which must be used in determining the radon-222 emissions from underground uranium mines, uranium mill tailings piles, phosphogypsum stacks, and other piles of waste material emitting radon.

1. Radon-222 Emissions from Underground Uranium Mine Vents

1.1 Sampling Frequency and Calculation of Emissions. Radon-222 emissions from underground uranium mine vents shall be determined using one of the following methods:

1.1.1 Continuous Measurement. These measurements shall be made and the emissions calculated as follows:

(a) The radon-222 concentration shall be continuously measured at each mine vent whenever the mine ventilation system is operational.

(b) Each mine vent exhaust flow rate shall be measured at least 4 times per year.

(c) A weekly radon-222 emission rate for the mine shall be calculated and recorded weekly as follows:

$$A_w = C_1 Q_1 T_1 + C_2 Q_2 T_2 + \dots C_n Q_n T_n$$

Where:

A_w = Total radon-222 emitted from the mine during week (Ci)

C_i = Average radon-222 concentration in mine vent i (Ci/m³)

Q_i = Volumetric flow rate from mine vent i (m³/hr)

T_i = Hours of mine ventilation system operation during week for mine vent i (hr)

(d) The annual radon-222 emission rate is the sum of the weekly emission rates during a calendar year.

1.1.2 Periodic Measurement. This method is applicable only to mines that continuously operate their ventilation system except for extended shutdowns. Mines which start up and shut down their ventilation system frequently must use the continuous measurement method describe in Section 1.1.1 above. Emission rates determined using periodic measurements shall be measured and calculated as follows:

(a) The radon-222 shall be continuously measured at each mine vent for at least one week every three months.

(b) Each mine vent exhaust flow rate shall be measured at least once during each of the radon-222 measurement periods.

(c) A weekly radon-222 emission rate shall be calculated for each weekly period accord-

ing to the method described in Section 1.1.1. In this calculation $T=168$ hr.

(d) The annual radon-222 emission rate from the mine should be calculated as follows:

$$A_y = \frac{52 - W_i}{n} (A_{w1} + A_{w2} + \dots A_{wn})$$

Where:

A_y = Annual radon-222 emission rate from the mine (Ci)

A_{wi} = Weekly radon-222 emission rate during the measurement period i (Ci)

n = Number of weekly measurement periods per year

W_i = Number of weeks during the year that the mine ventilation system is shut down in excess of 7 consecutive days, i.e. the sum of the number of weeks each shut down exceeds 7 days

1.2 Test Methods and Procedures

Each underground mine required to test its emissions, unless an equivalent or alternative method has been approved by the Administrator, shall use the following test methods:

1.2.1 Test Method 1 of Appendix A to part 60 shall be used to determine velocity traverses. The sampling point in the duct shall be either the centroid of the cross section or the point of average velocity.

1.2.2 Test Method 2 of Appendix A to part 60 shall be used to determine velocity and volumetric flow rates.

1.2.3 Test Methods A-6 or A-7 of Appendix B, Method 114 to part 61 shall be used for the analysis of radon-222. Use of Method A-7 requires prior approval of EPA based on conditions described in Appendix B.

1.2.4 A quality assurance program shall be conducted in conformance with the programs described for Continuous Radon Monitors and Alpha Track Detectors in EPA 520/1-89-009. (2)

2. Radon-222 Emissions from Uranium Mill Tailings Piles

2.1 Measurement and Calculation of Radon Flux from Uranium Mill Tailings Piles.

2.1.1 Frequency of Flux Measurement. A single set of radon flux measurements may be made, or if the owner or operator chooses, more frequent measurements may be made over a one year period. These measurements may involve quarterly, monthly or weekly intervals. All radon measurements shall be made as described in paragraphs 2.1.2 through 2.1.6 except that for measurements made over a one year period, the requirement of paragraph 2.1.4(c) shall not apply. The mean radon flux from the pile shall be the arithmetic mean of the mean

radon flux for each measurement period. The weather conditions, moisture content of the tailings and area of the pile covered by water existing at the time of the measurement shall be chosen so as to provide measurements representative of the long term radon flux from the pile and shall be subject to EPA review and approval.

2.1.2 Distribution of Flux Measurements. The distribution and number of radon flux measurements required on a pile will depend on clearly defined areas of the pile (called regions) that can have significantly different radon fluxes due to surface conditions. The mean radon flux shall be determined for each individual region of the pile. Regions that shall be considered for operating mill tailings piles are:

- (a) Water covered areas,
- (b) Water saturated areas (beaches),
- (c) Dry top surface areas, and
- (d) Sides, except where earthen material is used in dam construction.

For mill tailings after disposal the pile shall be considered to consist of only one region.

2.1.3 Number of Flux Measurements. Radon flux measurements shall be made within each region on the pile, except for those areas covered with water. Measurements shall be made at regularly spaced locations across the surface of the region, realizing that surface roughness will prohibit measurements in some areas of a region. The minimum number of flux measurements considered necessary to determine a representative mean radon flux value for each type of region on an operating pile is:

- (a) Water covered area—no measurements required as radon flux is assumed to be zero,
- (b) Water saturated beaches—100 radon flux measurements,
- (c) Loose and dry top surface—100 radon flux measurements,
- (d) Sides—100 radon flux measurements, except where earthen material is used in dam construction.

For a mill tailings pile after disposal which consists of only one region a minimum of 100 measurements are required.

2.1.4 Restrictions to Radon Flux Measurements. The following restrictions are placed on making radon flux measurements:

- (a) Measurements shall not be initiated within 24 hours of a rainfall.
- (b) If a rainfall occurs during the 24 hour measurements period, the measurement is invalid if the seal around the lip of the collector has washed away or if the collector is surrounded by water.
- (c) Measurements shall not be performed if the ambient temperature is below 35°F or if the ground is frozen.

2.1.5 Areas of Pile Regions. The approximate area of each region of the pile shall be determined in units of square meters.

2.1.6 Radon Flux Measurement. Measuring radon flux involves the adsorption of radon on activated charcoal in a large-area collector. The radon collector is placed on the surface of the pile area to be measured and allowed to collect radon for a time period of 24 hours. The radon collected on the charcoal is measured by gamma-ray spectroscopy. The detailed measurement procedure provided in Appendix A of EPA 520/5-85-0029(1) shall be used to measure the radon flux on uranium mill tailings, except the surface of the tailings shall not be penetrated by the lip of the radon collector as directed in the procedure, rather the collector shall be carefully positioned on a flat surface with soil or tailings used to seal the edge.

2.1.7 Calculations. The mean radon flux for each region of the pile and for the total pile shall be calculated and reported as follows:

- (a) The individual radon flux calculations shall be made as provided in Appendix A EPA 86 (1). The mean radon flux for each region of the pile shall be calculated by summing all individual flux measurements for the region and dividing by the total number of flux measurements for the region.
- (b) The mean radon flux for the total uranium mill tailings pile shall be calculated as follows.

$$J_t = \frac{J_1 A_1 + \dots + J_n A_n}{A_t}$$

Where:

J_t = Mean flux for the total pile (pCi/m²·s)

J_i = Mean flux measured in region i (pCi/m²·s)

A_i = Area of region i (m²)

A_t = Total area of the pile (m²)

2.1.8 Reporting. The results of individual flux measurements, the approximate locations on the pile, and the mean radon flux for the total stack shall be included in the emission test report. Any condition or unusual event that occurred during the measurements that could significantly affect the results should be reported.

3.0 Radon-222 Emissions from Phosphogypsum Stacks.

3.1 Measurement and Calculation of the Mean Radon Flux. Radon flux measurements shall be made on phosphogypsum stacks as described below:

3.1.1 Frequency of Measurements. A single set of radon flux measurements may be made after the phosphogypsum stack be-

comes inactive, or if the owner or operator chooses, more frequent measurements may be made over a one year period. These measurements may involve quarterly, monthly or weekly intervals. All radon measurements shall be made as described in paragraph 3.1.2 through 3.1.6 except that for measurements made over a one year period, the requirement of paragraph 3.1.4(c) shall not apply. For measurements made over a one year period, the radon flux shall be the arithmetic mean of the mean radon flux for each measurement period.

3.1.2 Distribution and Number of Flux Measurements. The distribution and number of radon flux measurements required on a stack will depend on clearly defined areas of the stack (called regions) that can have significantly different radon fluxes due to surface conditions. The mean radon flux shall be determined for each individual region of the stack. Regions that shall be considered are:

- (a) Water covered areas,
- (b) Water saturated areas (beaches),
- (c) Loose and dry top surface areas,
- (d) Hard-packed roadways, and
- (e) Sides.

3.1.3 Number of Flux Measurements. Radon flux measurements shall be made within each region on the phosphogypsum stack, except for those areas covered with water. Measurements shall be made at regularly spaced locations across the surface of the region, realizing that surface roughness will prohibit measurements in some areas of a region. The minimum number of flux measurements considered necessary to determine a representative mean radon flux value for each type of region is:

- (a) Water covered area—no measurements required as radon flux is assumed to be zero,
- (b) Water saturated beaches—50 radon flux measurements,
- (c) Loose and dry top surface—100 radon flux measurements,
- (d) Hard-packed roadways—50 radon flux measurements, and
- (e) Sides—100 radon flux measurements.

A minimum of 300 measurements are required. A stack that has no water cover can be considered to consist of two regions, top and sides, and will require a minimum of only 200 measurements.

3.1.4 Restrictions to Radon Flux Measurements. The following restrictions are placed on making radon flux measurements:

- (a) Measurements shall not be initiated within 24 hours of a rainfall.
- (b) If a rainfall occurs during the 24 hour measurement period, the measurement is invalid if the seal around the lip of the collector has washed away or if the collector is surrounded by water.

(c) Measurements shall not be performed if the ambient temperature is below 35 °F or if the ground is frozen.

3.1.5 Areas of Stack Regions. The approximate area of each region of the stack shall be determined in units of square meters.

3.1.6 Radon Flux Measurements. Measuring radon flux involves the adsorption of radon on activated charcoal in a large-area collector. The radon collector is placed on the surface of the stack area to be measured and allowed to collect radon for a time period of 24 hours. The radon collected on the charcoal is measured by gamma-ray spectroscopy. The detailed measurement procedure provided in Appendix A of EPA 520/5-85-0029(1) shall be used to measure the radon flux on phosphogypsum stacks, except the surface of the phosphogypsum

shall not be penetrated by the lip of the radon collector as directed in the procedure, rather the collector shall be carefully positioned on a flat surface with soil or phosphogypsum used to seal the edge.

3.1.7 Calculations. The mean radon flux for each region of the phosphogypsum stack and for the total stack shall be calculated and reported as follows:

- (a) The individual radon flux calculations shall be made as provided in Appendix A EPA 86 (1). The mean radon flux for each region of the stack shall be calculated by summing all individual flux measurements for the region and dividing by the total number of flux measurements for the region.
- (b) The mean radon flux for the total phosphogypsum stack shall be calculated as follows.

$$J_t = \frac{J_1 A_1 + J_2 A_2 + \dots + J_i A_i}{A_t}$$

Where:

J_t = Mean flux for the total stack (pCi/m²-s)

J_i = Mean flux measured in region i (pCi/m²-s)

A_i = Area of region i (m²)

A_t = Total area of the stack

3.1.8 Reporting. The results of individual flux measurements, the approximate locations on the stack, and the mean radon flux for each region and the mean radon flux for the total stack shall be included in the emission test report. Any condition or unusual event that occurred during the measurements that could significantly affect the results should be reported.

4.0 Quality Assurance Procedures for Measuring Rn-222 Flux

A. Sampling Procedures

Records of field activities and laboratory measurements shall be maintained. The following information shall be recorded for each charcoal canister measurement:

- (a) Site
- (b) Name of pile
- (c) Sample location
- (d) Sample ID number
- (e) Date and time on
- (f) Date and time off
- (g) Observations of meteorological conditions and comments

Records shall include all applicable information associated with determining the sample measurement, calculations, observations, and comments.

B. Sample Custody

Custodial control of all charcoal samples exposed in the field shall be maintained in

accordance with EPA chain-of-custody field procedures. A control record shall document all custody changes that occur between the field and laboratory personnel.

C. Calibration Procedures and Frequency

The radioactivity of two standard charcoal sources, each containing a carefully determined quantity of radium-226 uniformly distributed through 180g of activated charcoal, shall be measured. An efficiency factor is computed by dividing the average measured radioactivity of the two standard charcoal sources, minus the background, in cpm by the known radioactivity of the charcoal sources in dpm. The same two standard charcoal sources shall be counted at the beginning and at the end of each day's counting as a check of the radioactivity counting equipment. A background count using unexposed charcoal should also be made at the beginning and at the end of each counting day to check for inadvertent contamination of the detector or other changes affecting the background. The unexposed charcoal comprising the blank is changed with each new batch of charcoal used.

D. Internal Quality Control Checks and Frequency

The charcoal from every tenth exposed canister shall be recounted. Five percent of the samples analyzed shall be either blanks (charcoal having no radioactivity added) or samples spiked with known quantities of radium-226.

E. Data Precision, Accuracy, and Completeness

The precision, accuracy, and completeness of measurements and analyses shall be

within the following limits for samples measuring greater than $1.0 \text{ pCi/m}^3 - \text{s}$.

- (a) Precision: 10%
- (b) Accuracy: $\pm 10\%$
- (c) Completeness: at least 85% of the measurements must yield useable results.

5.0 References

(1) Hartley, J.N. and Freeman, H.D., "Radon Flux Measurements on Gardiner and Royster Phosphogypsum Piles Near Tampa and Mulberry, Florida," U.S. Environmental Protection Agency Report, EPA 520/5-85-029, January 1986.

(2) Environmental Protection Agency, "Indoor Radon and Radon Decay Product Measurement Protocols", EPA 520/1-89-009, U.S. Environmental Protection Agency, Washington, DC. (1989).

[38 FR 8826, Apr. 6, 1973]

EDITORIAL NOTE: For FEDERAL REGISTER citations to Appendix B see the List of CFR Sections Affected appearing in the Finding Aids section of this volume.

APPENDIX C—QUALITY ASSURANCE PROCEDURES

Procedure 1—Determination of Adequate Chromatographic Peak Resolution

In this method of dealing with resolution, the extent to which one chromatographic peak overlaps another is determined.

For convenience, consider the range of the elution curve of each compound as running from -2σ to $+2\sigma$. This range is used in other resolution criteria, and it contains 95.45 percent of the area of a normal curve. If two peaks are separated by a known distance, b , one can determine the fraction of the area of one curve that lies within the range of the other. The extent to which the elution curve of a contaminant compound overlaps the curve of a compound that is under analysis is found by integrating the contaminant curve over the limits $b-2\sigma$ to $b+2\sigma$, where σ , is the standard deviation of the sample curve.

This calculation can be simplified in several ways. Overlap can be determined for curves of unit area; then actual areas can be introduced. Desired integration can be resolved into two integrals of the normal distribution function for which there are convenient calculation programs and tables. An example would be Program 15 in Texas Instruments Program Manual ST1, 1975, Texas Instruments, Inc., Dallas, Texas 75222.

$$\frac{1}{\sqrt{2\pi}\sigma_s} \int_{b-2\sigma_s}^{b+2\sigma_s} e^{\left(\frac{-t^2}{2\sigma_s^2}\right)} dt = \frac{1}{\sqrt{2\pi}} \int_{\frac{b-2\sigma_s}{\sigma_c}}^{\infty} e^{\left(\frac{-x^2}{2}\right)} dx - \frac{1}{\sqrt{2\pi}} \int_{\frac{b+2\sigma_s}{\sigma_c}}^{\infty} e^{\left(\frac{-x^2}{2}\right)} dx$$

The following calculation steps are required:*

1. $2\sigma_s = t_s / \sqrt{2 \ln 2}$
2. $\sigma_c = t_c / \sqrt{2 \ln 2}$
3. $x_1 = (b - 2\sigma_s) / \sigma_c$
4. $x_2 = (b + 2\sigma_s) / \sigma_c$
5. $Q(x_1) = \frac{1}{\sqrt{2\pi}} \int_{x_1}^{\infty} e^{\left(\frac{-x^2}{2}\right)} dx$
6. $Q(x_2) = \frac{1}{\sqrt{2\pi}} \int_{x_2}^{\infty} e^{\left(\frac{-x^2}{2}\right)} dx$

7. $I_o = Q(x_1) - Q(x_2)$
8. $A_o = I_o A_c / A_s$
9. Percentage overlap = $A_o \times 100$.

where:

A_s = Area of the sample peak of interest determined by electronic integration or by the formula $A_s = h_s t_s$.

A_c = Area of the contaminant peak, determined in the same manner as A_s .

b = Distance on the chromatographic chart that separates the maxima of the two peaks.

h_s = Peak height of the sample compound of interest, measured from the average value of the baseline to the maximum of the curve.

t_s = Width of sample peak of interest at 1/2 peak height.

t_c = Width of the contaminant peak at 1/2 of peak height.

σ_s = Standard deviation of the sample compound of interest elution curve.

σ_c = Standard deviation of the contaminant elution curve.

$Q(x_1)$ = Integral of the normal distribution function from x_1 to infinity.

$Q(x_2)$ = Integral of the normal distribution function from x_2 to infinity.

I_o = Overlap integral.

A_o = Area overlap fraction.

*In most instances, $Q(x_2)$ is very small and may be neglected.

In judging the suitability of alternate GC columns or the effects of altering chromatographic conditions, one can employ the area overlap as the resolution parameter with a specific maximum permissible value.

The use of Gaussian functions to describe chromatographic elution curves is widespread. However, some elution curves are highly asymmetric. In cases where the sample peak is followed by a contaminant that has a leading edge that rises sharply but the curve then tails off, it may be possible to define an effective width for t_r as "twice the distance from the leading edge to a perpendicular line through the maxim of the contaminant curve, measured along a perpendicular bisection of that line."

Procedure 2—Procedure for Field Auditing GC Analysis

Responsibilities of audit supervisor and analyst at the source sampling site include the following:

A. The audit supervisor verifies that audit cylinders are stored in a safe location both before and after the audit to prevent vandalism.

B. At the beginning and conclusion of the audit, the analyst records each cylinder number and pressure. An audit cylinder is never analyzed when the pressure drops below 200 psi.

C. During the audit, the analyst performs a minimum of two consecutive analyses of each audit cylinder gas. The audit must be conducted to coincide with the analysis of source test samples, normally immediately after GC calibration and prior to sample analyses.

D. At the end of audit analyses, the audit supervisor requests the calculated concentrations from the analyst and compares the results with the actual audit concentrations. If each measured concentration agrees with the respective actual concentration within ± 10 percent, he directs the analyst to begin analyzing source samples. Audit supervisor judgment and/or supervisory policy determine action when agreement is not within ± 10 percent. When a consistent bias in excess of 10 percent is found, it may be possible to proceed with the sample analysis, with a corrective factor to be applied to the results at a later time. However, every attempt should be made to locate the cause of the discrepancy, as it may be misleading. The audit supervisor records each cylinder number, cylinder pressure (at the end of the audit), and all calculated concentrations. The individual being audited must not under any circumstance be told actual audit concentrations until calculated concentrations have been submitted to the audit supervisor.

FIELD AUDIT REPORT

Part A.—To be filled out by organization supplying audit cylinders.

1. Organization supplying audit sample(s) and shipping address _____

2. Audit supervisor, organization, and phone number _____

3. Shipping instructions: Name, Address, Attention _____

4. Guaranteed arrival date for cylinders _____

5. Planned shipping date for cylinders _____

6. Details on audit cylinders from last analysis _____

	Low conc.	High conc.
a. Date of last analysis.....		
b. Cylinder number.....		
c. Cylinder pressure, psi.....		
d. Audit gas(es)/balance gas.....		
e. Audit gas(es), ppm.....		
f. Cylinder construction.....		

Part B.—To be filled out by audit supervisor.

1. Process sampled _____

2. Audit location _____

3. Name of individual audit _____

4. Audit date _____

5. Audit results:

	Low conc. cylinder	High conc. cylinder
a. Cylinder number.....		
b. Cylinder pressure before audit, psi.....		
c. Cylinder pressure after audit, psi.....		
d. Measured concentration, ppm injection #1* Injection #2* Average.....		
e. Actual audit concentration, ppm (Part A, 6e).....		
f. Audit accuracy: ¹ Low Conc. Cylinder..... High Conc. Cylinder..... Percent ¹ accuracy =		
Measured Conc. - Actual Conc. _____ × 100 Actual Conc.		
g. Problems detected (if any).....		

Results of two consecutive injections that meet the sample analysis criteria of the test method.

[47 FR 39178, Sept. 7, 1982]

APPENDIX D TO PART 61—METHODS FOR ESTIMATING RADIONUCLIDE EMISSIONS

1. Purpose and Background

Facility owners or operators may estimate radionuclide emissions to the atmosphere for dose calculations instead of measuring emissions. Particulate emissions from mill tailings piles should be estimated using the procedures listed in reference #2. All other emissions may be estimated by using the "Procedures" listed below, or using the method described in reference #1.

2. Procedure

To estimate emissions to the atmosphere:

(a) Determine the amount (in curies) used at facilities for the period under consideration. Radioactive materials in sealed packages that remain unopened, and have not leaked during the assessment period should not be included in the calculation.

(b) Multiply the amount used by the following factors which depend on the physical state of the radionuclide. They are:

- (i) 1 for gases;
- (ii) 10^{-3} for liquids or particulate solids; and
- (iii) 10^{-6} for solids.

If any nuclide is heated to a temperature of 100 degrees Celsius or more, boils at a temperature of 100 degrees Celsius or less, or is intentionally dispersed into the environment, it must be considered to be a gas.

(c) If a control device is installed between the place of use and the point of release, multiply emissions from (b) by an adjustment factor. These are presented in Table 1.

TABLE 1—ADJUSTMENT TO EMISSION FACTORS FOR EFFLUENT CONTROLS

Controls	Types of radionuclides controlled	Adjustment factor to emissions	Comments and conditions
HEPA filters	Particulates	0.01	Not applicable to gaseous radionuclides; periodic testing is prudent to ensure high removal efficiency.
Fabric filter	Particulates	0.1	Monitoring would be prudent to guard against tears in filter.
Sintered metal	Particulates	1	Insufficient data to make recommendation.
Activated carbon filters	Iodine gas	0.1	Efficiency is time dependent; monitoring is necessary to ensure effectiveness.
Douglas bags: Held one week or longer for decay	Xenon	0.5/wk	Based on xenon half-life of 5.3 days;
Douglas bags: Released within one week	Xenon	1	Provides no reduction of exposure to general public.
Venturi scrubbers	Particulates	0.05	Although venturis may remove gases, variability in gaseous removal efficiency dictates adjustment factor for particulates only.
	Gases	1	
Packed bed scrubbers	Gases	0.1	Not applicable to particulates.
Electrostatic precipitators	Particulates	0.05	Not applicable for gaseous radionuclides.
Xenon traps	Xenon	0.1	Efficiency is time dependent; monitoring is necessary to ensure effectiveness.
Fume hoods	All	1	Provides no reduction to general public exposures.
Vent stacks	All	1	Generally provides no reduction of exposure to general public.

References

(1) Environmental Protection Agency, "A Guide for Determining Compliance with the Clean Air Act Standards for Radionuclides Emissions from NRC-Licensed and Non-DOE Federal Facilities", EPA 520/1-89-002, January 1989.

(2) Nuclear Regulatory Commission, "Methods for Estimating Radioactive and Toxic Airborne Source Terms for Uranium Milling Operations", U.S. Nuclear Regulatory Commission Regulatory Guide 3.59, March 1987.

[54 FR 51711, Dec. 15, 1989]

APPENDIX E TO PART 61—COMPLIANCE PROCEDURES METHODS FOR DETERMINING COMPLIANCE WITH SUBPART I

1. Purpose and Background

This Appendix provides simplified procedures to reduce the burden on Nuclear Regulatory Commission (NRC) licensees, and non-Department of Energy Federal facilities in determining compliance with 40 CFR part 61, subpart I. The procedures consist of

a series of increasingly more stringent steps, depending on the facility's potential to exceed the standard.

First, a facility can be found in compliance if the quantity of radioactive material possessed during the year is less than that listed in a table of annual possession quantities. A facility will also be in compliance if the average annual radionuclide emission concentration is less than that listed in a table of air concentration levels. If the facility is not in compliance by these tables, it can establish compliance by estimating a dose using screening procedure developed by the National Council on Radiation Protection and Measurements with a radiological source term derived using EPA approved emission factors. These procedures are described in a "Guide for Determining Compliance with the Clean Air Act Standards for Radionuclide Emissions From NRC-Licensed and Non-DOE Federal Facilities."

A user-friendly computer program called COMPLY has been developed to reduce the burden on the regulated community. The Agency has also prepared a "User's Guide for the COMPLY Code" to assist the regulated community in using the code, and in handling more complex situations such as multiple release points. The basis for these compliance procedures are provided in "Background Information Document: Procedures Approved for Demonstrating Compliance with 40 CFR part 61, subpart I". The compliance model is the highest level in the COMPLY computer code and provides for the most realistic assessment of dose by allowing the use of site-specific information.

2. Table of Annual Possession Quantity

(a) Table 1 may be used for determining if facilities are in compliance with the standard. The possession table can only be used if the following conditions are met:

(i) No person lives within 10 meters of any release point; and

(ii) No milk, meat, or vegetables are produced within 100 meters of any release point.

(b) Procedures described in Reference (1) shall be used to determine compliance or exemption from reporting by use of Table 2.

TABLE 1—ANNUAL POSSESSION QUANTITIES FOR ENVIRONMENTAL COMPLIANCE

[Annual Possession Quantities (Ci/yr)]

Radionuclide	Gas- ous form*	Liquid/ powder forms	Solid form*
Ac-225	9.6E-05	9.6E-02	9.6E+01
Ac-227	1.6E-07	1.6E-04	1.6E-01
Ac-228	3.4E-03	3.4E+00	3.4E+03
Ag-106	1.6E+00	1.6E+03	1.6E+06

TABLE 1—ANNUAL POSSESSION QUANTITIES FOR ENVIRONMENTAL COMPLIANCE—Continued

[Annual Possession Quantities (Ci/yr)]

Radionuclide	Gas- ous form*	Liquid/ powder forms	Sol- id**
Ag-106m	2.6E-03	2.6E+00	2.6E+03
Ag-108m	6.5E-06	6.5E-03	6.5E+00
Ag-110m	9.4E-05	9.4E-02	9.4E+01
Ag-111	6.7E-02	6.7E+01	6.7E+04
Al-26	4.0E-06	4.0E-03	4.0E+00
Am-241	2.3E-06	2.3E-03	2.3E+00
Am-242	1.8E-02	1.8E+01	1.8E+04
Am-242m	2.5E-06	2.5E-03	2.5E+00
Am-243	2.3E-06	2.3E-03	2.3E+00
Am-244	4.6E-02	4.6E+01	4.6E+04
Am-245	7.0E+00	7.0E+03	7.0E+06
Am-246	9.8E-01	9.8E+02	9.8E+05
Ar-37	1.4E+06		
Ar-41	1.4E+00		
As-72	2.9E-02	2.9E+01	2.9E+04
As-73	6.0E-02	6.0E+01	6.0E+04
As-74	4.3E-03	4.3E+00	4.3E+03
As-76	8.8E-02	8.8E+01	8.8E+04
As-77	7.9E-01	7.9E+02	7.9E+05
At-211	1.0E-02	1.0E+01	1.0E+04
Au-193	4.2E-01	4.2E+02	4.2E+05
Au-194	3.5E-02	3.5E+01	3.5E+04
Au-195	3.3E-03	3.3E+00	3.3E+03
Au-198	4.6E-02	4.6E+01	4.6E+04
Au-199	1.5E-01	1.5E+02	1.5E+05
Ba-131	1.0E-02	1.0E+01	1.0E+04
Ba-133	4.9E-05	4.9E-02	4.9E+01
Ba-133m	9.3E-02	9.3E+01	9.3E+04
Ba-135m	5.8E-01	5.8E+02	5.8E+05
Ba-139	4.7E+00	4.7E+03	4.7E+06
Ba-140	2.1E-03	2.1E+00	2.1E+03
Ba-141	1.3E+00	1.3E+03	1.3E+06
Ba-142	1.1E+00	1.1E+03	1.1E+06
Be-7	2.3E-02	2.3E+01	2.3E+04
Be-10	3.0E-03	3.0E+00	3.0E+03
Bi-206	3.1E-03	3.1E+00	3.1E+03
Bi-207	8.4E-06	8.4E-03	8.4E-00
Bi-210	4.2E-03	4.2E+00	4.2E+03
Bi-212	4.7E-02	4.7E+01	4.7E+04
Bi-213	6.0E-02	6.0E+01	6.0E+04
Bi-214	1.4E-01	1.4E+02	1.4E+05
Bk-249	7.0E-04	7.0E-01	7.0E+02
Bk-250	1.0E-01	1.0E+02	1.0E+05
Br-77	7.5E-02	7.5E+01	7.5E+04
Br-80	1.2E+01	1.2E+04	1.2E+07
Br-80m	1.5E+00	1.5E+03	1.5E+06
Br-82	1.6E-02	1.6E+01	1.6E+04
Br-83	9.9E+00	9.9E+03	9.9E+06
Br-84	5.6E-01	5.6E+02	5.6E+05
C-11	1.3E+00	1.3E+03	1.3E+06
C-14	2.9E-01	2.9E+02	2.9E+05
Ca-41	2.7E-02	2.7E+01	2.7E+04
Ca-45	5.8E-02	5.8E+01	5.8E+04
Ca-47	1.1E-02	1.1E+01	1.1E+04
Cd-109	5.0E-03	5.0E+00	5.0E+03
Cd-113	3.3E-04	3.3E-01	3.3E+02
Cd-113m	4.4E-04	4.4E-01	4.4E+02
Cd-115	5.4E-02	5.4E+01	5.4E+04
Cd-115m	1.0E-02	1.0E+01	1.0E+04
Cd-117	5.6E-02	5.6E+01	5.6E+04
Cd-117m	1.3E-01	1.3E+02	1.3E+05
Ce-139	2.6E-03	2.6E+00	2.6E+03
Ce-141	1.8E-02	1.8E+01	1.8E+04
Ce-143	1.0E-01	1.0E+02	1.0E+05
Ce-144	1.7E-03	1.7E+00	1.7E+03
Cf-248	2.0E-05	2.0E-02	2.0E+01

TABLE 1—ANNUAL POSSESSION QUANTITIES
FOR ENVIRONMENTAL COMPLIANCE—Continued

[Annual Possession Quantities (Ci/yr)]

Radionuclide	Gas- ous form*	Liquid/ powder forms	Solid form*
Ci-249	1.7E-06	1.7E-03	1.7E+00
Ci-250	4.0E-06	4.0E-03	4.0E+00
Ci-251	1.7E-06	1.7E-03	1.7E+00
Ci-252	6.4E-06	6.4E-03	6.4E+00
Ci-253	3.3E-04	3.3E-01	3.3E+02
Ci-254	3.6E-06	3.6E-03	3.6E+00
Ci-26	1.9E-04	1.9E-01	1.9E+02
Ci-38	6.5E-01	6.5E+02	6.5E+05
Cm-242	6.0E-05	6.0E-02	6.0E+01
Cm-243	3.3E-06	3.3E-03	3.3E+00
Cm-244	4.2E-06	4.2E-03	4.2E+00
Cm-245	2.3E-06	2.3E-03	2.3E+00
Cm-246	2.3E-06	2.3E-03	2.3E+00
Cm-247	2.3E-06	2.3E-03	2.3E+00
Cm-248	6.4E-07	6.4E-04	6.4E-01
Cm-249	4.6E+00	4.6E+03	4.6E+06
Cm-250	1.1E-07	1.1E-04	1.1E-01
Co-56	2.4E-04	2.4E-01	2.4E+02
Co-57	1.6E-03	1.6E+00	1.6E+03
Co-58	9.0E-04	9.0E-01	9.0E+02
Co-58m	1.7E-01	1.7E+02	1.7E+05
Co-60	1.6E-05	1.6E-02	1.6E+01
Co-60m	4.0E+00	4.0E+03	4.0E+06
Co-61	3.8E+00	3.8E+03	3.8E+06
Co-69	9.0E-01	9.0E+02	9.0E+05
Co-61	6.3E-02	6.3E+01	6.3E+04
Cs-129	1.5E-01	1.5E+02	1.5E+05
Cs-131	2.8E-01	2.8E+02	2.8E+05
Cs-132	1.3E-02	1.3E+01	1.3E+04
Cs-134	5.2E-05	5.2E-02	5.2E+01
Cs-134m	3.2E-01	3.2E+02	3.2E+05
Cs-135	2.4E-02	2.4E+01	2.4E+04
Cs-136	2.1E-03	2.1E+00	2.1E+03
Cs-137	2.3E-05	2.3E-02	2.3E+01
Cs-138	4.4E-01	4.4E+02	4.4E+05
Cu-61	4.0E-01	4.0E+02	4.0E+05
Cu-64	5.2E-01	5.2E+02	5.2E+05
Co-67	1.5E-01	1.5E+02	1.5E+05
Dy-157	4.4E-01	4.4E+02	4.4E+05
Dy-165	5.6E+00	5.6E+03	5.6E+06
Dy-166	8.1E-02	8.1E+01	8.1E+04
Er-169	4.0E-01	4.0E+02	4.0E+05
Er-171	3.6E-01	3.6E+02	3.6E+05
Es-253	2.6E-04	2.6E-01	2.6E+02
Es-254	2.3E-05	2.3E-02	2.3E+01
Es-254m	1.8E-03	1.8E+00	1.8E+03
Eu-152	1.6E-05	1.6E-02	1.6E+01
Eu-152m	3.5E-01	3.5E+02	3.5E+05
Eu-154	2.0E-05	2.0E-02	2.0E+01
Eu-155	5.2E-04	5.2E-01	5.2E+02
Eu-156	3.2E-03	3.2E+00	3.2E+03
F-18	5.6E-01	5.6E+02	5.6E+05
Fe-52	4.9E-02	4.9E+01	4.9E+04
Fe-55	1.4E-01	1.4E+02	1.4E+05
Fe-59	1.3E-03	1.3E+00	1.3E+03
Fm-254	1.8E-02	1.8E+01	1.8E+04
Fm-255	4.0E-03	4.0E+00	4.0E+03
Fr-223	1.4E-01	1.4E+02	1.4E+05
Ga-66	5.6E-02	5.6E+01	5.6E+04
Ga-67	1.1E-01	1.1E+02	1.1E+05
Ga-68	7.6E-01	7.6E+02	7.6E+05
Gd-152	3.6E-02	3.6E+01	3.6E+04
Gd-153	4.4E-06	4.4E-03	4.4E+00
Gd-159	2.0E-03	2.0E+00	2.0E+03
Ge-68	6.8E-01	6.8E+02	6.8E+05
	2.3E-04	2.3E-01	2.3E+02

TABLE 1—ANNUAL POSSESSION QUANTITIES
FOR ENVIRONMENTAL COMPLIANCE—Continued

[Annual Possession Quantities (Ci/yr)]

Radionuclide	Gas- ous form*	Liquid/ powder forms	Solid form*
Ge-71	2.6E+00	2.6E+03	2.6E+06
Ge-77	1.0E-01	1.0E+02	1.0E+05
H-3	1.5E+01	1.5E+04	1.5E+07
Hi-181	2.5E-03	2.5E+00	2.5E+03
Hg-193m	9.5E-02	9.5E+01	9.5E+04
Hg-197	2.4E-01	2.4E+02	2.4E+05
Hg-197m	2.5E-01	2.5E+02	2.5E+05
Hg-203	5.2E-03	5.2E+00	5.2E+03
Ho-166	2.8E-01	2.8E+02	2.8E+05
Ho-166m	6.0E-06	6.0E-03	6.0E+00
I-123	4.9E-01	4.9E+02	4.9E+05
I-124	9.3E-03	9.3E+00	9.3E+03
I-125	6.2E-03	6.2E+00	6.2E+03
I-126	3.7E-03	3.7E+00	3.7E+03
I-128	9.3E+00	9.3E+03	9.3E+06
I-129	2.6E-04	2.6E-01	2.6E+02
I-130	4.6E-02	4.6E+01	4.6E+04
I-131	6.7E-03	6.7E+00	6.7E+03
I-132	2.0E-01	2.0E+02	2.0E+05
I-133	6.7E-02	6.7E+01	6.7E+04
I-134	3.2E-01	3.2E+02	3.2E+05
I-135	1.2E-01	1.2E+02	1.2E+05
In-111	4.9E-02	4.9E+01	4.9E+04
In-113m	2.1E+00	2.1E+03	2.1E+06
In-114m	4.9E-03	4.9E+00	4.9E+03
In-115	2.7E-04	2.7E-01	2.7E+02
In-115m	1.4E+00	1.4E+03	1.4E+06
In-116m	3.5E-01	3.5E+02	3.5E+05
In-117	1.3E+00	1.3E+03	1.3E+06
In-117m	7.6E-02	7.6E+01	7.6E+04
Ir-190	3.5E-03	3.5E+00	3.5E+03
Ir-192	9.7E-04	9.7E-01	9.7E+02
Ir-194	2.5E-01	2.5E+02	2.5E+05
Ir-194m	1.5E-04	1.5E-01	1.5E+02
K-40	6.8E-05	6.8E-02	6.8E+01
K-42	2.9E-01	2.9E+02	2.9E+05
K-43	6.0E-02	6.0E+01	6.0E+04
K-44	4.9E-01	4.9E+02	4.9E+05
Kr-79	7.0E+00		
Kr-81	1.8E+02		
Kr-83m	2.0E+04		
Kr-85	8.4E+02		
Kr-85m	1.1E+01		
Kr-87	2.0E+00		
Kr-88	4.2E-01		
La-140	1.6E-02	1.6E+01	1.6E+04
La-141	1.1E+00	1.1E+03	1.1E+06
La-142	2.3E-01	2.3E+02	2.3E+05
Lu-177	1.4E-01	1.4E+02	1.4E+05
Lu-177m	3.5E-04	3.5E-01	3.5E+02
Mg-28	2.1E-02	2.1E+01	2.1E+04
Mn-52	3.5E-03	3.5E+00	3.5E+03
Mn-52m	5.2E-01	5.2E+02	5.2E+05
Mn-53	5.7E-02	5.7E+01	5.7E+04
Mn-54	2.5E-04	2.5E-01	2.5E+02
Mn-56	2.5E-01	2.5E+02	2.5E+05
Mo-93	1.5E-03	1.5E+00	1.5E+03
Mo-99**	5.7E-02	5.7E+01	5.7E+04
Mo-101	8.4E-01	8.4E+02	8.4E+05
Na-22	3.2E-05	3.2E-02	3.2E+01
Na-24	2.6E-02	2.6E+01	2.6E+04
Nb-90	2.5E-02	2.5E+01	2.5E+04
Nb-93m	1.2E-02	1.2E+01	1.2E+04
Nb-94	6.0E-06	6.0E-03	6.0E+00
Nb-95	2.3E-03	2.3E+00	2.3E+03
Nb-95m	2.0E-02	2.0E+01	2.0E+04

TABLE 1—ANNUAL POSSESSION QUANTITIES
FOR ENVIRONMENTAL COMPLIANCE—Continued

(Annual Possession Quantities (Ci/yr))

Radionuclide	Gas- ous form*	Liquid/ powder forms	Solid form*
Nb-96	2.5E-02	2.5E+01	2.5E+04
Nb-97	1.0E+00	1.0E+03	1.0E+06
Nd-147	3.0E-02	3.0E+01	3.0E+04
Nd-149	1.1E+00	1.1E+03	1.1E+06
Ni-56	2.0E-03	2.0E+00	2.0E+03
Ni-57	2.1E-02	2.1E+01	2.1E+04
Ni-59	2.2E-02	2.2E+01	2.2E+04
Ni-63	1.4E-01	1.4E+02	1.4E+05
Ni-65	7.0E-01	7.0E+02	7.0E+05
Np-235	3.0E-02	3.0E+01	3.0E+04
Np-237	1.8E-06	1.8E-03	1.8E+00
Np-238	1.9E-02	1.9E+01	1.9E+04
Np-239	1.0E-01	1.0E+02	1.0E+05
Np-240	6.5E-01	6.5E+02	6.5E+05
Np-240m	4.7E+00	4.7E+03	4.7E+06
Os-185	9.2E-04	9.2E-01	9.2E+02
Os-191m	9.0E-01	9.0E+02	9.0E+05
Os-191	3.8E-02	3.8E+01	3.8E+04
Os-193	2.9E-01	2.9E+02	2.9E+05
P-32	1.7E-02	1.7E+01	1.7E+04
P-33	1.2E-01	1.2E+02	1.2E+05
Pa-230	6.3E-04	6.3E-01	6.3E+02
Pa-231	8.3E-07	8.3E-04	8.3E-01
Pa-233	9.3E-03	9.3E+00	9.3E+03
Pa-234	9.3E-02	9.3E+01	9.3E+04
Pb-203	8.3E-02	8.3E+01	8.3E+04
Pb-205	1.2E-02	1.2E+01	1.2E+04
Pb-209	1.1E+01	1.1E+04	1.1E+07
Pb-210	5.5E-05	5.5E-02	5.5E+01
Pb-211	1.2E-01	1.2E+02	1.2E+05
Pb-212	6.0E-03	6.0E+00	6.0E+03
Pb-214	1.2E-01	1.2E+02	1.2E+05
Pd-103	2.1E-01	2.1E+02	2.1E+05
Pd-107	8.2E-02	8.2E+01	8.2E+04
Pd-109	9.4E-01	9.4E+02	9.4E+05
Pm-143	7.6E-04	7.6E-01	7.6E+02
Pm-144	1.1E-04	1.1E-01	1.1E+02
Pm-145	5.2E-04	5.2E-01	5.2E+02
Pm-146	4.4E-05	4.4E-02	4.4E+01
Pm-147	2.6E-02	2.6E+01	2.6E+04
Pm-148	1.7E-02	1.7E+01	1.7E+04
Pm-148m	7.6E-04	7.6E-01	7.6E+02
Pm-149	2.8E-01	2.8E+02	2.8E+05
Pm-151	1.2E-01	1.2E+02	1.2E+05
Po-210	9.3E-05	9.3E-02	9.3E+01
Pr-142	2.8E-01	2.8E+02	2.8E+05
Pr-143	1.0E-01	1.0E+02	1.0E+05
Pr-144	1.5E+01	1.5E+04	1.5E+07
Pt-191	6.4E-02	6.4E+01	6.4E+04
Pt-193	2.1E-02	2.1E+01	2.1E+04
Pt-193m	4.8E-01	4.8E+02	4.8E+05
Pt-195m	1.4E-01	1.4E+02	1.4E+05
Pt-197	1.1E+00	1.1E+03	1.1E+06
Pt-197m	3.6E+00	3.6E+03	3.6E+06
Pu-236	7.0E-06	7.0E-03	7.0E+00
Pu-237	2.3E-02	2.3E+01	2.3E+04
Pu-238	2.7E-06	2.7E-03	2.7E+00
Pu-239	2.5E-06	2.5E-03	2.5E+00
Pu-240	2.5E-06	2.5E-03	2.5E+00
Pu-241	1.3E-04	1.3E-01	1.3E+02
Pu-242	2.5E-06	2.5E-03	2.5E+00
Pu-243	3.8E+00	3.8E+03	3.8E+06
Pu-244	2.4E-06	2.4E-03	2.4E+00
Pu-245	2.1E-01	2.1E+02	2.1E+05
Pu-246	4.8E-03	4.8E+00	4.8E+03
Ra-223	1.3E-04	1.3E-01	1.3E+02

TABLE 1—ANNUAL POSSESSION QUANTITIES
FOR ENVIRONMENTAL COMPLIANCE—Continued

(Annual Possession Quantities (Ci/yr))

Radionuclide	Gas- ous form*	Liquid/ powder forms	Solid form*
Ra-224	3.2E-04	3.2E-01	3.2E+02
Ra-225	1.3E-04	1.3E-01	1.3E+02
Ra-226	5.5E-06	5.5E-03	5.5E+00
Ra-228	1.3E-05	1.3E-02	1.3E+01
Rb-81	4.2E-01	4.2E+02	4.2E+05
Rb-83	1.4E-03	1.4E+00	1.4E+03
Rb-84	2.0E-03	2.0E+00	2.0E+03
Rb-86	1.7E-02	1.7E+01	1.7E+04
Rb-87	1.0E-02	1.0E+01	1.0E+04
Rb-88	1.7E+00	1.7E+03	1.7E+06
Rb-89	6.4E-01	6.4E+02	6.4E+05
Re-184	1.8E-03	1.8E+00	1.8E+03
Re-184m	3.6E-04	3.6E-01	3.6E+02
Re-186	1.9E-01	1.9E+02	1.9E+05
Re-187	9.3E+00	9.3E+03	9.3E+06
Re-188	3.7E-01	3.7E+02	3.7E+05
Rh-103m	1.7E+02	1.7E+05	1.7E+08
Rh-105	3.4E-01	3.4E+02	3.4E+05
Ru-97	8.3E-02	8.3E+01	8.3E+04
Ru-103	3.1E-03	3.1E+00	3.1E+03
Ru-105	2.9E-01	2.9E+02	2.9E+05
Ru-106	5.9E-04	5.9E-01	5.9E+02
S-35	7.5E-02	7.5E+01	7.5E+04
Sb-117	2.0E+00	2.0E+03	2.0E+06
Sb-122	3.9E-02	3.9E+01	3.9E+04
Sb-124	6.0E-04	6.0E-01	6.0E+02
Sb-125	1.4E-04	1.4E-01	1.4E+02
Sb-126	1.8E-03	1.8E+00	1.8E+03
Sb-126m	7.6E-01	7.6E+02	7.6E+05
Sb-127	2.0E-02	2.0E+01	2.0E+04
Sb-129	1.8E-01	1.8E+02	1.8E+05
Sc-44	1.4E-01	1.4E+02	1.4E+05
Sc-46	4.0E-04	4.0E-01	4.0E+02
Sc-47	1.1E-01	1.1E+02	1.1E+05
Sc-48	1.1E-02	1.1E+01	1.1E+04
Sc-49	1.0E-01	1.0E+04	1.0E+07
Se-73	1.6E-01	1.6E+02	1.6E+05
Se-75	1.1E-03	1.1E+00	1.1E+03
Se-79	6.9E-03	6.9E+00	6.9E+03
Si-31	4.7E+00	4.7E+03	4.7E+06
Si-32	7.2E-04	7.2E-01	7.2E+02
Sm-147	1.4E-05	1.4E-02	1.4E+01
Sm-151	3.5E-02	3.5E+01	3.5E+04
Sm-153	2.4E-01	2.4E+02	2.4E+05
Sn-113	1.9E-03	1.9E+00	1.9E+03
Sn-117m	2.3E-02	2.3E+01	2.3E+04
Sn-119m	2.9E-02	2.9E+01	2.9E+04
Sn-123	1.8E-02	1.8E+01	1.8E+04
Sn-125	7.2E-03	7.2E+00	7.2E+03
Sn-126	4.7E-06	4.7E-03	4.7E+00
Sr-82	1.9E-03	1.9E+00	1.9E+03
Sr-85	1.9E-03	1.9E+00	1.9E+03
Sr-85m	1.5E+00	1.5E+03	1.5E+06
Sr-87m	1.2E+00	1.2E+03	1.2E+06
Sr-89	2.1E-02	2.1E+01	2.1E+04
Sr-90	5.2E-04	5.2E-01	5.2E+02
Sr-91	1.2E-01	1.2E+02	1.2E+05
Sr-92	2.5E-01	2.5E+02	2.5E+05
Ta-182	4.4E-04	4.4E-01	4.4E+02
Tb-157	2.2E-03	2.2E+00	2.2E+03
Tb-160	8.4E-04	8.4E-01	8.4E+02
Tc-95	9.0E-02	9.0E+01	9.0E+04
Tc-95m	1.4E-03	1.4E+00	1.4E+03
Tc-96	5.6E-03	5.6E+00	5.6E+03
Tc-96m	7.0E-01	7.0E+02	7.0E+05
Tc-97	1.5E-03	1.5E+00	1.5E+03

TABLE 1—ANNUAL POSSESSION QUANTITIES
FOR ENVIRONMENTAL COMPLIANCE—Continued

[Annual Possession Quantities (Ci/yr)]

Radionuclide	Gaseous form*	Liquid/powder forms	Solid form*
Tc-97m	7.2E-02	7.2E+01	7.2E+04
Tc-98	6.4E-06	6.4E-03	6.4E+00
Tc-99	9.0E-03	9.0E+00	9.0E+03
Tc-99m	1.4E+00	1.4E+03	1.4E+06
Tc-101	3.8E+00	3.8E+03	3.8E+06
Tc-121	6.0E-03	6.0E+00	6.0E+03
Tc-121m	5.3E-04	5.3E-01	5.3E+02
Tc-123	1.2E-03	1.2E+00	1.2E+03
Tc-123m	2.7E-03	2.7E+00	2.7E+03
Tc-125m	1.5E-02	1.5E+01	1.5E+04
Tc-127	2.9E+00	2.9E+03	2.9E+06
Tc-127m	7.3E-03	7.3E+00	7.3E+03
Tc-129	6.5E+00	6.5E+03	6.5E+06
Tc-129m	6.1E-03	6.1E+00	6.1E+03
Tc-131	9.4E-01	9.4E+02	9.4E+05
Tc-131m	1.8E-02	1.8E+01	1.8E+04
Tc-132	6.2E-03	6.2E+00	6.2E+03
Tc-133	1.2E+00	1.2E+03	1.2E+06
Tc-133m	2.9E-01	2.9E+02	2.9E+05
Tc-134	4.4E-01	4.4E+02	4.4E+05
Th-226	3.0E-02	3.0E+01	3.0E+04
Th-227	6.4E-05	6.4E-02	6.4E+01
Th-228	2.9E-06	2.9E-03	2.9E+00
Th-229	4.9E-07	4.9E-04	4.9E-01
Th-230	3.2E-06	3.2E-03	3.2E+00
Th-231	8.4E-01	8.4E+02	8.4E+05
Th-232	6.0E-07	6.0E-04	6.0E-01
Th-234	2.0E-02	2.0E+01	2.0E+04
Th-234m	5.2E-06	5.2E-03	5.2E+00
Th-235	4.0E-01	4.0E+02	4.0E+05
Th-235m	4.4E-02	4.4E+01	4.4E+04
Th-236	1.8E-01	1.8E+02	1.8E+05
Th-236m	1.0E-02	1.0E+01	1.0E+04
Th-237	2.5E-02	2.5E+01	2.5E+04
Th-237m	2.4E-02	2.4E+01	2.4E+04
Th-238	5.9E-02	5.9E+01	5.9E+04
Th-238m	5.0E-05	5.0E-02	5.0E+01
Th-239	1.4E-01	1.4E+02	1.4E+05
Th-239m	1.3E-06	1.3E-03	1.3E+00
Th-240	7.6E-06	7.6E-03	7.6E+00
Th-240m	7.6E-06	7.6E-03	7.6E+00
Th-241	7.0E-06	7.0E-03	7.0E+00
Th-241m	8.4E-06	8.4E-03	8.4E+00
Th-242	4.7E-02	4.7E+01	4.7E+04
Th-242m	8.6E-06	8.6E-03	8.6E+00
Th-243	8.3E+00	8.3E+03	8.3E+06
Th-244	1.8E-01	1.8E+02	1.8E+05
Th-244m	1.4E-03	1.4E+00	1.4E+03
Th-245	1.3E+00	1.3E+03	1.3E+06
Th-245m	1.1E-02	1.1E+01	1.1E+04
Th-246	1.6E-01	1.6E+02	1.6E+05
Th-246m	1.1E-01	1.1E+02	1.1E+05
Th-247	1.0E-02	1.0E+01	1.0E+04
Th-247m	7.6E-02	7.6E+01	7.6E+04
Th-248	1.6E+00	1.6E+03	1.6E+06
Th-248m	6.0E-01		
Th-249	7.0E+00		
Th-249m	7.6E+01		
Th-250	2.2E+02		
Th-250m	5.2E+01		
Th-251	6.0E+01		
Th-251m	7.6E+00		
Th-252	4.2E+00		
Th-252m	9.9E-01		
Th-253	2.8E-02	2.8E+01	2.8E+04
Th-253m	2.3E-02	2.3E+01	2.3E+04

TABLE 1—ANNUAL POSSESSION QUANTITIES
FOR ENVIRONMENTAL COMPLIANCE—Continued

[Annual Possession Quantities (Ci/yr)]

Radionuclide	Gaseous form*	Liquid/powder forms	Solid form*
Y-88	2.5E-04	2.5E-01	2.5E+02
Y-90	1.1E-01	1.1E+02	1.1E+05
Y-90m	4.3E-01	4.3E+02	4.3E+05
Y-91	1.8E-02	1.8E+01	1.8E+04
Y-91m	1.6E+00	1.6E+03	1.6E+06
Y-92	7.0E-01	7.0E+02	7.0E+05
Y-93	3.8E-01	3.8E+02	3.8E+05
Yb-169	5.5E-03	5.5E+00	5.5E+03
Yb-175	2.1E-01	2.1E+02	2.1E+05
Zn-62	8.6E-02	8.6E+01	8.6E+04
Zn-65	4.4E-04	4.4E-01	4.4E+02
Zn-69	2.7E+01	2.7E+04	2.7E+07
Zn-69m	2.0E-01	2.0E+02	2.0E+05
Zr-86	2.4E-02	2.4E+01	2.4E+04
Zr-88	2.7E-04	2.7E-01	2.7E+02
Zr-89	1.6E-02	1.6E+01	1.6E+04
Zr-93	2.8E-03	2.8E+00	2.8E+03
Zr-95	6.4E-04	6.4E-01	6.4E+02
Zr-97	4.6E-02	4.6E+01	4.6E+04

*Radionuclides boiling at 100 °C or less, or exposed to a temperature of 100 °C, must be considered a gas. Capsules containing radionuclides in liquid or powder form can be considered to be solids.

**Mo-99 contained in a generator to produce Technetium-99m can be assumed to be a solid.

3. Table of Concentration Levels

(a) Table 2 may be used for determining if facilities are in compliance with the standard.

1. The concentration table as applied to emission estimates can only be used if all releases are from point sources and concentrations have been measured at the stack or vent using EPA-approved methods, and the distance between each stack or vent and the nearest resident is greater than 3 times the diameter of the stack or vent. Procedures provided in Ref. (1) shall be used to determine compliance or exemption from reporting by use of Table 2.

2. The concentration table may be used to determine compliance with the standard based on environmental measurements provided these measurements are made in conformance with the requirements of § 61.107(b)(5).

4. NCRP Screening Model

The procedures described in Reference (4) may be used to determine doses to members of the general public from emissions of radionuclides to the atmosphere. Both the total dose from all radionuclides emitted, and the dose caused by radioactive iodine must be considered in accordance with the procedures in Ref. (1).

5. The COMPLY Computer Code

The COMPLY computer code may be used to determine compliance with subpart I. The compliance model in the COMPLY computer code may be used to determine the dose to members of the general public from emissions of radionuclides to the atmosphere. The EPA may add radionuclides to all or any part of COMPLY to cover radionuclides that may be used by the regulated community.

TABLE 2—CONCENTRATION LEVELS FOR ENVIRONMENTAL COMPLIANCE

Radionuclide	Concentration (Ci/m ³)	Radionuclide	Concentration (Ci/m ³)
Ac-225	9.1E-14	Bi-207	1.0E-14
Ac-227	1.0E-16	Bi-210	2.9E-13
Ac-228	3.7E-12	Bi-212	5.6E-11
Ag-106	1.9E-09	Bi-213	7.1E-11
Ag-106m	1.2E-12	Bi-214	1.4E-10
Ag-108m	7.1E-15	Bk-249	5.6E-13
Ag-110m	9.1E-14	Bk-250	9.1E-11
Ag-111	2.5E-12	Br-77	4.2E-11
Al-26	4.8E-15	Br-80	1.4E-08
Am-241	1.9E-15	Br-80m	1.8E-09
Am-242	1.5E-11	Br-82	1.2E-11
Am-242m	2.0E-15	Br-83	1.2E-08
Am-243	1.8E-15	Br-84	6.7E-10
Am-244	4.0E-11	C-11	1.5E-09
Am-245	8.3E-09	C-14	1.0E-11
Am-246	1.2E-09	Ca-41	4.2E-13
Ar-37	1.6E-03	Ca-45	1.3E-12
Ar-41	1.7E-09	Ca-47	2.4E-12
As-72	2.4E-11	Cd-109	5.9E-13
As-73	1.1E-11	Cd-113	9.1E-15
As-74	2.2E-12	Cd-113m	1.7E-14
As-76	5.0E-11	Cd-115	1.6E-11
As-77	1.6E-10	Cd-115m	8.3E-13
At-211	1.1E-11	Cd-117	6.7E-11
Au-193	3.8E-10	Cd-117m	1.6E-10
Au-194	3.2E-11	Ce-139	2.6E-12
Au-195	3.1E-12	Ce-141	6.3E-12
Au-198	2.1E-11	Ce-143	3.0E-11
Au-199	4.8E-11	Ce-144	6.2E-13
Ba-131	7.1E-12	Cf-243	1.8E-14
Ba-133	3.9E-14	Cf-249	1.4E-15
Ba-133m	5.9E-11	Cf-250	3.2E-15
Ba-135m	1.8E-10	Cf-251	1.4E-15
Ba-139	5.6E-09	Cf-252	5.6E-15
Ba-140	1.3E-12	Cf-253	3.1E-13
Ba-141	1.4E-09	Cf-254	3.0E-15
Ba-142	1.3E-09	Cf-36	2.7E-15
Be-7	2.3E-11	Ci-38	7.7E-10
Be-10	1.6E-12	Cm-242	5.3E-14
Bi-206	2.3E-12	Cm-243	2.6E-15
Cm-244	3.3E-15	Eu-156	1.9E-12
Cm-245	1.8E-15	F-18	6.7E-10
Cm-246	1.9E-15	Fe-52	5.6E-11
Cm-247	1.9E-15	Fe-55	9.1E-12
Cm-248	5.0E-16	Fe-59	6.7E-13
Cm-249	3.7E-09	Fm-254	2.0E-11
Cm-250	9.1E-17	Fm-255	4.3E-12
Co-56	1.8E-13	Fr-223	3.3E-11
Co-57	1.3E-12	Ga-66	6.2E-11
Co-58	6.7E-13	Ga-67	7.1E-11
Co-58m	1.2E-10	Ga-68	9.1E-10
Co-60	1.7E-14	Ga-72	3.8E-11
Co-60m	4.3E-09	Gd-152	5.0E-15

TABLE 2—CONCENTRATION LEVELS FOR ENVIRONMENTAL COMPLIANCE—Continued

Radionuclide	Concentration (Ci/m ³)	Radionuclide	Concentration (Ci/m ³)
Co-61	4.5E-09	Gd-153	2.1E-12
Cr-49	1.1E-09	Gd-159	2.9E-12
Cr-51	3.1E-11	Ge-68	2.0E-12
Cs-129	1.4E-10	Ge-71	2.4E-12
Cs-131	3.3E-11	Ge-77	1.0E-12
Cs-132	4.8E-12	H-3	1.5E-28
Cs-134	2.7E-14	Hf-181	1.9E-12
Cs-134m	1.7E-10	Hg-193m	1.0E-12
Cs-135	4.0E-13	Hg-197	8.3E-12
Cs-136	5.3E-13	Hg-197m	1.0E-12
Cs-137	1.9E-14	Hg-203	1.1E-12
Cs-138	5.3E-10	Ho-166	7.1E-11
Cu-61	4.8E-10	Ho-166m	7.1E-15
Cu-64	5.3E-10	I-123	4.3E-12
Cu-67	5.0E-11	I-124	6.2E-12
Dy-157	5.0E-10	I-125	1.2E-12
Dy-165	6.7E-09	I-126	1.1E-12
Dy-166	1.1E-11	I-128	1.1E-28
Er-169	2.9E-11	I-129	9.1E-15
Er-171	4.0E-10	I-130	4.5E-11
Es-253	2.4E-13	I-131	2.1E-12
Es-254	2.0E-14	I-132	2.3E-12
Es-254m	1.8E-12	I-133	2.0E-12
Eu-152	2.0E-14	I-134	3.8E-12
Eu-152m	3.6E-10	I-135	1.2E-12
Eu-154	2.3E-14	In-111	3.8E-12
Eu-155	5.9E-13	In-113m	2.5E-28
In-114m	1.9E-13	Nb-95	2.2E-13
In-115	7.1E-14	Nb-95m	1.4E-12
In-115m	1.6E-09	Nb-96	2.4E-12
In-116m	4.2E-10	Nb-97	1.2E-28
In-117	1.6E-09	Nd-147	7.7E-12
In-117m	9.1E-11	Nd-149	7.1E-12
Ir-190	2.6E-12	Ni-56	1.7E-12
Ir-192	9.1E-13	Ni-57	1.8E-12
Ir-194	1.1E-10	Ni-59	1.5E-12
Ir-194m	1.7E-13	Ni-63	1.4E-12
K-40	2.7E-14	Ni-65	8.3E-12
K-42	2.6E-10	Np-235	2.5E-12
K-43	6.2E-11	Np-237	1.2E-12
K-44	5.9E-10	Np-238	1.4E-12
Kr-79	8.3E-09	Np-239	3.9E-12
Kr-81	2.1E-07	Np-240	7.7E-12
Kr-83m	2.3E-05	Np-240m	5.8E-28
Kr-85	1.0E-06	Os-185	1.0E-12
Kr-85m	1.3E-08	Os-191m	2.9E-12
Kr-87	2.4E-09	Os-191	1.1E-12
Kr-88	5.0E-10	Os-193	9.1E-12
La-140	1.2E-11	P-32	3.3E-12
La-141	7.7E-10	P-33	2.4E-12
La-142	2.7E-10	Pa-230	3.2E-12
Lu-177	2.4E-11	Pa-231	5.9E-12
Lu-177m	3.6E-13	Pa-233	4.0E-12
Mg-28	1.5E-11	Pa-234	1.1E-12
Mn-52	2.8E-12	Pb-203	6.2E-12
Mn-52m	6.2E-10	Pb-205	5.6E-12
Mn-53	1.5E-11	Pb-209	1.3E-28
Mn-54	2.8E-13	Pb-210	2.8E-15
Mn-56	2.9E-10	Pb-211	1.4E-12
Mo-93	1.1E-12	Pb-212	6.3E-12
Mo-99	1.4E-11	Pb-214	1.2E-12
Mo-101	1.0E-09	Pd-103	3.8E-12
Na-22	2.6E-14	Pd-107	3.1E-12
Na-24	2.6E-11	Pd-109	4.8E-12
Nb-90	1.6E-11	Pm-143	9.1E-12
Nb-93m	2.0E-11	Pm-144	1.3E-12
Nb-94	7.1E-15	Pm-145	6.2E-12

TABLE 2—CONCENTRATION LEVELS FOR ENVIRONMENTAL COMPLIANCE—Continued

Radionuclide	Concentration (Ci/m ³)	Radionuclide	Concentration (Ci/m ³)
Pm-146	5.3E-14	Re-184m	3.7E-13
Pm-147	1.1E-11	Re-186	1.8E-11
Pm-148	5.0E-12	Re-187	2.6E-10
Pm-148m	6.7E-13	Re-188	1.7E-10
Pm-149	4.2E-11	Rh-103m	2.1E-07
Pm-151	7.1E-11	Rh-105	1.3E-10
Po-210	7.1E-15	Ru-97	6.7E-11
Po-142	1.1E-10	Ru-103	2.6E-12
Po-143	7.1E-12	Ru-105	2.8E-10
Po-144	1.8E-08	Ru-106	3.4E-13
Pl-101	4.3E-11	S-35	1.3E-12
Pl-193	1.8E-11	Sb-117	2.4E-09
Pl-193m	4.8E-11	Sb-122	1.4E-11
Pl-135m	3.2E-11	Sb-124	5.3E-13
Pl-197	4.0E-10	Sb-125	1.6E-13
Pl-197m	2.6E-09	Sb-126	1.4E-12
Pu-236	5.9E-15	Sb-126m	9.1E-10
Pu-237	1.9E-11	Sb-127	7.1E-12
Pu-238	2.1E-15	Sb-129	7.7E-11
Pu-239	2.0E-15	Sc-44	1.7E-10
Pu-240	2.0E-15	Sc-46	4.2E-13
Pu-241	1.0E-13	Sc-47	3.8E-11
Pu-242	2.0E-15	Sc-48	9.1E-12
Pu-243	4.2E-09	Sc-49	1.2E-08
Pu-244	2.0E-15	Se-73	1.7E-10
Pu-245	2.1E-10	Se-75	1.7E-13
Pu-246	2.2E-12	Se-79	1.1E-13
Ra-223	4.2E-14	Si-31	5.6E-09
Ra-224	1.5E-13	Si-32	3.4E-14
Ra-225	5.0E-14	Sm-147	1.4E-14
Ra-226	3.3E-15	Sm-151	2.1E-11
Ra-228	5.9E-15	Sm-153	5.9E-11
Rb-81	5.0E-10	Sn-113	1.4E-12
Rb-83	3.4E-13	Sn-117m	5.6E-12
Rb-84	3.6E-13	Sn-119m	5.3E-12
Rb-86	5.6E-13	Sn-123	1.1E-12
Rb-87	1.6E-13	Sn-125	1.7E-12
Rb-88	2.1E-09	Sn-126	5.3E-15
Rb-89	7.1E-10	Sr-82	6.2E-13
Re-184	1.5E-12	Sr-85	1.8E-12
Sr-85m	1.6E-09	Th-232	6.2E-16
Sr-87m	1.4E-09	Th-234	2.2E-12
Sr-89	1.8E-12	Ti-44	6.2E-15
Sr-90	1.9E-14	Ti-45	4.8E-10
Sr-91	9.1E-11	Ti-200	4.5E-11
Sr-92	2.9E-10	Ti-201	1.0E-10
Ta-182	4.5E-13	Ti-202	5.0E-12
Tb-157	2.5E-12	Ti-204	1.2E-12
Tb-160	7.7E-13	Tm-170	3.3E-12
Tc-95	1.0E-10	Tm-171	2.6E-11
Tc-95m	1.4E-12	U-230	1.5E-14
Tc-96	5.6E-12	U-231	4.2E-11
Tc-96m	6.7E-10	U-232	1.3E-15
Tc-97	7.1E-13	U-233	7.1E-15
Tc-97m	7.1E-12	U-234	7.7E-15
Tc-98	6.7E-15	U-235	7.1E-15
Tc-99	1.4E-13	U-236	7.7E-15
Tc-99m	1.7E-09	U-237	1.0E-11
Tc-101	4.5E-09	U-238	8.3E-15
Tc-121	1.0E-12	U-239	4.3E-09
Tc-121m	1.2E-13	U-240	1.3E-10
Tc-123	1.4E-13	V-48	1.0E-12
Tc-123m	2.0E-13	V-49	1.6E-10
Tc-125m	3.6E-13	W-181	6.7E-12
Tc-127	1.0E-09	W-185	2.6E-12
Tc-127m	1.5E-13	W-187	7.7E-11
Tc-129	7.7E-09	W-188	5.3E-13

TABLE 2—CONCENTRATION LEVELS FOR ENVIRONMENTAL COMPLIANCE—Continued

Radionuclide	Concentration (Ci/m ³)	Radionuclide	Concentration (Ci/m ³)
Te-129m	1.4E-13	Xe-122	9.1E-11
Te-131	9.1E-11	Xe-123	1.6E-09
Te-131m	1.0E-12	Xe-125	1.1E-11
Te-132	7.1E-13	Xe-127	8.3E-09
Te-133	9.1E-10	Xe-129m	9.1E-08
Te-133m	2.2E-10	Xe-131m	2.6E-07
Te-134	5.3E-10	Xe-133	6.2E-08
Th-226	3.4E-11	Xe-133m	7.1E-08
Th-227	3.8E-14	Xe-135	9.1E-09
Th-228	3.1E-15	Xe-135m	5.0E-09
Th-229	5.3E-16	Xe-138	1.2E-09
Th-230	3.4E-15	Y-86	3.0E-11
Th-231	2.9E-10	Y-87	1.7E-11
Y-88	2.7E-13	Zn-65	9.1E-14
Y-90	1.3E-11	Zn-69	3.2E-08
Y-90m	1.9E-10	Zn-69m	1.7E-10
Y-91	2.1E-12	Zr-86	2.4E-11
Y-91m	1.3E-09	Zr-88	3.1E-13
Y-92	8.3E-10	Zr-89	1.3E-11
Y-93	2.9E-10	Zr-93	2.6E-12
Yb-169	3.7E-12	Zr-95	6.7E-13
Yb-175	4.3E-11	Zr-97	3.8E-11
Zn-62	9.1E-11		

6. References

- (1) Environmental Protection Agency, "A Guide for Determining Compliance with the Clean Air Act Standards for Radionuclides Emissions from NRC-Licensed and Non-DOE Federal Facilities", EPA 520/1-89-002, October 1989.
- (2) Environmental Protection Agency, "User's Guide for the COMPLY Code", EPA 520/1-89-003, October 1989.
- (3) Environmental Protection Agency, "Background Information Document: Procedures Approved for Demonstrating Compliance with 40 CFR part 61, subpart I", EPA 520/1-89-001, January 1989.
- (4) National Council on Radiation Protection and Measurement, "Screening Techniques for Determining Compliance with Environmental Standards" NCRP Commentary No. 3, Revision of January 1989 with addendum of October, 1989.

[54 FR 51711, Dec. 15, 1989]

PART 62—APPROVAL AND PROMULGATION OF STATE PLANS FOR DESIGNATED FACILITIES AND POLLUTANTS

Subpart A—General Provisions

- Sec.
- 62.01 Definitions.
 - 62.02 Introduction.
 - 62.03 Extensions.
 - 62.04 Approval status.
 - 62.05 Legal authority.
 - 62.06 Negative declarations.
 - 62.07 Emissions standards, compliance schedules.
 - 62.08 Emission inventories and source surveillance.
 - 62.09 Revision of plans by Administrator.
 - 62.10 Submission to Administrator.
 - 62.11 Severability.
 - 62.12 Availability of applicable plans.

Subpart B—Alabama

PLAN FOR THE CONTROL OF DESIGNATED POLLUTANTS FROM EXISTING FACILITIES (SECTION 111(d) PLAN)

- 62.100 Identification of plan.

SULFURIC ACID MIST FROM EXISTING SULFURIC ACID PLANTS

- 62.101 Identification of sources.

FLUORIDE EMISSIONS FROM PHOSPHATE FERTILIZER PLANTS

- 62.102 Identification of sources.

Subpart C—Alaska

FLUORIDE EMISSIONS FROM PHOSPHATE FERTILIZER PLANTS

- 62.350 Identification of plan—negative declaration.

ACID MIST FROM SULFURIC ACID PLANTS

- 62.351 Identification of plan—negative declaration.

TOTAL REDUCED SULFUR EMISSIONS FROM KRAFT PULP MILLS

- 62.352 Identification of plan—negative declaration.

FLUORIDE EMISSIONS FROM PRIMARY ALUMINUM REDUCTION PLANTS

- 62.353 Identification of plan—negative declaration.

Subpart D—[Reserved]

Subpart E—Arkansas

PLAN FOR THE CONTROL OF DESIGNATED POLLUTANTS FROM EXISTING FACILITIES (SECTION 111(d) PLAN)

- 62.850 Identification of plan.
- 62.852 Emission inventories, source surveillance, reports.

FLUORIDE EMISSIONS FROM EXISTING PHOSPHATE FERTILIZER PLANTS

- 62.854 Identification of sources.

SULFURIC ACID MIST EMISSIONS FROM EXISTING SULFURIC ACID PLANTS

- 62.855 Identification of sources.

TOTAL REDUCED SULPHUR EMISSIONS FROM EXISTING KRAFT PULP MILLS

- 62.865 Identification of sources.
- 62.866 Compliance schedule.

Subpart F—Plan for the Control of Designated Pollutants From Existing Facilities (Section 111(d) Plan)

- 62.1100 Identification of plan.

FLUORIDE EMISSIONS FROM EXISTING PHOSPHATE FERTILIZER PLANTS

- 62.1101 Identification of sources.

SULFURIC ACID MIST EMISSIONS FROM EXISTING SULFURIC ACID PRODUCTION UNITS

- 62.1102 Identification of sources.

FLUORIDE EMISSIONS FROM PRIMARY ALUMINUM REDUCTION PLANTS

- 62.1103 Identification of plan—negative declaration.

TOTAL REDUCED SULPHUR EMISSIONS FROM EXISTING KRAFT PULP MILLS

- 62.1104 Identification of sources.

Subpart G—[Reserved]

Subpart H—Connecticut

FLUORIDE EMISSIONS FROM PHOSPHATE FERTILIZER PLANTS

- 62.1600 Identification of plan—negative declaration.

SULFURIC ACID MIST EMISSIONS FROM SULFURIC ACID PRODUCTION UNITS

- 62.1625 Identification of plan—negative declaration. 4Total Reduced Sulfur Emissions From Existing Kraft Pulp Mills

Environmental Protection Agency

Part 62

TOTAL REDUCED SULFUR EMISSIONS FROM EXISTING KRAFT PULP MILLS

62.1850 Identification of plan—negative declaration.

FLUORIDE EMISSIONS FROM EXISTING PRIMARY ALUMINUM PLANTS

62.1700 Identification of plan—negative declaration.

Subpart I—Delaware

FLUORIDE EMISSIONS FROM PHOSPHATE FERTILIZER PLANTS

62.1850 Identification of plan—negative declaration.

SULFURIC ACID MIST FROM EXISTING SULFURIC ACID PLANTS

62.1875 Identification of plan.

TOTAL REDUCED SULFUR EMISSIONS FROM EXISTING KRAFT PULP MILLS

62.1900 Identification—negative declaration.

FLUORIDE EMISSIONS FROM PRIMARY ALUMINUM REDUCTION PLANTS

62.1925 Identification of plan—negative declaration.

Subpart J—District of Columbia

FLUORIDE EMISSIONS FROM PHOSPHATE FERTILIZER PLANTS

62.2100 Identification of plan—negative declaration.

SULFURIC ACID MIST EMISSIONS FROM EXISTING SULFURIC ACID PLANTS

62.2101 Identification of plan—negative declaration.

TOTAL REDUCED SULFUR EMISSIONS FROM EXISTING KRAFT PULP MILLS

62.2110 Identification of plan—negative declaration.

FLUORIDE EMISSIONS FROM EXISTING PRIMARY ALUMINUM PLANTS

62.2120 Identification of plan—negative declaration.

Subpart K—Florida

PLAN FOR THE CONTROL OF DESIGNATED POLLUTANTS FROM EXISTING FACILITIES (SECTION 111(d) PLAN)

62.2350 Identification of plan.

SULFURIC ACID MIST FROM EXISTING SULFURIC ACID PLANTS

62.2351 Identification of sources.

FLUORIDE EMISSIONS FROM PRIMARY ALUMINUM REDUCTION PLANTS

62.2352 Identification of source—negative declaration.

TOTAL REDUCED SULFUR EMISSIONS FROM KRAFT PULP MILLS AND TALL OIL PLANTS

62.2353 Identification of sources.

62.2354 Compliance schedules.

Subpart L—Georgia

PLAN FOR THE CONTROL OF DESIGNATED POLLUTANTS FROM EXISTING FACILITIES (SECTION 111(d) PLAN)

62.2600 Identification of plan.

SULFURIC ACID MIST FROM EXISTING SULFURIC ACID PLANTS

62.2601 Identification of sources.

FLUORIDE EMISSIONS FROM PHOSPHATE FERTILIZER PLANTS

62.2602 Identification of sources—Negative declaration.

TOTAL REDUCED SULFUR EMISSIONS FROM KRAFT PULP MILLS

62.2603 Identification of sources.

62.2604 Compliance schedules.

FLUORIDE EMISSIONS FROM EXISTING PRIMARY ALUMINUM REDUCTION PLANTS

62.2605 Identification of sources—Negative declaration.

Subpart M—[Reserved]

Subpart N—Idaho

FLUORIDE EMISSIONS FROM EXISTING PRIMARY ALUMINUM PLANTS

62.3100 Identification of plan—negative declaration.

Subpart O—Illinois

SULFURIC ACID MIST EMISSIONS FROM EXISTING SULFURIC ACID PRODUCTION PLANTS

62.3300 Identification of plan.

TOTAL REDUCED SULFUR EMISSIONS FROM KRAFT PULP MILLS

62.3325 Identification of plan—negative declaration.

Subpart P—Indiana

FLUORIDE EMISSIONS FROM PHOSPHATE FERTILIZER PLANTS

62.3600 Identification of plan—negative declaration.

FLUORIDE EMISSIONS FROM EXISTING
PRIMARY ALUMINUM PLANTS

62.3625 Identification of plan.

Subpart Q—Iowa

PLAN FOR THE CONTROL OF DESIGNATED POL-
LUTANTS FROM EXISTING FACILITIES (SEC-
TION 111(d) PLAN)

62.3850 Identification of plan.

SULFURIC ACID MIST FROM EXISTING
SULFURIC ACID PRODUCTION PLANTS

62.3851 Identification of sources.

FLUORIDE EMISSIONS FROM EXISTING
PHOSPHATE FERTILIZER PLANTS

62.3852 Identification of source.

TOTAL REDUCED SULFUR EMISSIONS FROM
EXISTING KRAFT PULP MILLS

62.3853 Identification of plan—negative
declaration.

FLUORIDE EMISSIONS FROM EXISTING
PRIMARY ALUMINUM REDUCTION PLANTS

62.3854 Identification of plan—negative
declaration.

TOTAL REDUCED SULFUR EMISSIONS FROM
EXISTING KRAFT PULP MILLS

62.3910 Identification of plan—negative
declaration.

Subpart R—Kansas

FLUORIDE EMISSIONS FROM EXISTING
PHOSPHATE FERTILIZER PLANTS

62.4100 Identification of plan—negative
declaration.

TOTAL REDUCED SULFUR EMISSIONS FROM
EXISTING KRAFT PULP MILLS

62.4125 Identification of plan—negative
declaration.

FLUORIDE EMISSIONS FROM EXISTING
PRIMARY ALUMINUM REDUCTION PLANTS

62.4150 Identification of plan—negative
declaration.

SULFURIC ACID MIST FROM EXISTING
SULFURIC ACID PRODUCTION PLANTS

62.4175 Identification of plan.

Subpart S—Kentucky

PLAN FOR THE CONTROL OF DESIGNATED POL-
LUTANTS FROM EXISTING FACILITIES (SEC-
TION 111(d) PLAN)

62.4350 Identification of plan.

SULFURIC ACID MIST FROM EXISTING
SULFURIC ACID PLANTS

62.4351 Identification of sources.

TOTAL REDUCED SULFUR FROM EXISTING
KRAFT PULP MILLS

62.4352 Identification of sources.

FLUORIDE EMISSIONS FROM EXISTING
PRIMARY ALUMINUM REDUCTION PLANTS

62.4353 Identification of sources.

FLUORIDE EMISSIONS FROM PHOSPHATE
FERTILIZER PLANTS

62.4354 Identification of plan—negative
declaration.

Subpart T—Louisiana

PLAN FOR CONTROL OF DESIGNATED POLLU-
TANTS FROM EXISTING FACILITIES (SEC-
TION 111(d) PLAN)

62.4620 Identification of plan.

62.4621 Emission standards and compli-
ance schedules.

62.4622 Emission inventories, source su-
veillance, reports.

62.4623 Legal authority.

SULFURIC ACID MIST FROM EXISTING
SULFURIC ACID PLANTS

62.4624 Identification of sources.

FLUORIDE EMISSIONS FROM EXISTING
PHOSPHATE FERTILIZER PLANTS

62.4625 Identification of sources.

62.4626 Effective date.

FLUORIDE EMISSIONS FROM EXISTING
PRIMARY ALUMINUM PLANTS

62.4627 Identification of sources.

62.4628 Effective date.

TOTAL REDUCED SULFUR EMISSIONS FROM
EXISTING KRAFT PULP MILLS

62.4629 Identification of sources.

62.4630 Effective date.

Subpart U—Maine

PLAN FOR THE CONTROL OF DESIGNATED POL-
LUTANTS FROM EXISTING FACILITIES (SEC-
TION 111(d) PLAN)

62.4845 Identification of plan.

FLUORIDE EMISSIONS FROM EXISTING
PRIMARY ALUMINUM PLANTS

62.4875 Identification of sources—negative
declaration.

SULFURIC ACID MIST FROM EXISTING
SULFURIC ACID PLANTS

62.4900 Identification of sources.

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FLUORIDE EMISSIONS FROM PHOSPHATE FERTILIZER PLANTS

62.4950 Identification of plan—negative declaration.

Subpart V—Maryland

PLAN FOR CONTROL OF DESIGNATED POLLUTANTS FROM EXISTING FACILITIES (SECTION 111(d) PLAN)

62.5100 Identification of plan.

SULFURIC ACID MIST FROM EXISTING SULFURIC ACID PLANTS

62.5101 Identification of sources.

TOTAL REDUCED SULFUR EMISSIONS FROM EXISTING KRAFT PULP MILLS

62.5102 Identification of sources.

FLUORIDE EMISSIONS FROM PRIMARY ALUMINUM PLANTS

62.5103 Identification of sources.

Subpart W—Massachusetts

FLUORIDE EMISSIONS FROM PHOSPHATE FERTILIZER PLANTS

62.5350 Identification of plan—negative declaration.

SULFURIC ACID MIST EMISSIONS FROM EXISTING SULFURIC ACID PLANTS

62.5351 Identification of plan—negative declaration.

TOTAL REDUCED SULFUR EMISSIONS FROM EXISTING KRAFT PULP MILLS

62.5375 Identification of plan—negative declaration.

FLUORIDE EMISSIONS FROM EXISTING PRIMARY ALUMINUM PLANTS

62.5400 Identification of plan—negative declaration.

Subpart X—Michigan

FLUORIDE EMISSIONS FROM PHOSPHATE FERTILIZER PLANTS

62.5600 Identification of plan—negative declaration.

Subpart Y—Minnesota

FLUORIDE EMISSIONS FROM PHOSPHATE FERTILIZER PLANTS

62.5850 Identification of plan—negative declaration.

Subpart Z—Mississippi

PLAN FOR THE CONTROL OF DESIGNATED POLLUTANTS FROM EXISTING FACILITIES (SECTION 111(d) PLAN)

62.6100 Identification of plan.

SULFURIC ACID MIST FROM EXISTING SULFURIC ACID PLANTS

62.6110 Identification of sources.

FLUORIDE EMISSIONS FROM PHOSPHATE FERTILIZER PLANTS

62.6120 Identification of sources.

FLUORIDE EMISSIONS FROM PRIMARY ALUMINUM PLANTS

62.6121 Identification of sources—negative declaration.

TOTAL REDUCED SULFUR EMISSIONS FROM KRAFT PULP MILLS

62.6122 Identification of sources.

Subpart AA—Missouri

PLAN FOR THE CONTROL OF DESIGNATED POLLUTANTS FROM EXISTING FACILITIES (SECTION 111(d) PLAN)

62.6350 Identification of plan.

FLUORIDE EMISSIONS FROM EXISTING PHOSPHATE FERTILIZER PLANTS

62.6351 Identification of sources.

FLUORIDE EMISSIONS FROM EXISTING¹ PRIMARY ALUMINUM REDUCTION PLANTS

62.6352 Identification of sources.

SULFURIC ACID MIST FROM EXISTING SULFURIC ACID PRODUCTION PLANTS

62.6353 Identification of sources.

TOTAL REDUCED SULFUR EMISSIONS FROM EXISTING KRAFT PULP MILLS

62.6354 Identification of plan—negative declaration.

Subpart BB—[Reserved]

Subpart CC—Nebraska

FLUORIDE EMISSIONS FROM EXISTING PHOSPHATE FERTILIZER PLANTS

62.6850 Identification of plan—negative declaration.

SULFURIC ACID MIST EMISSIONS FROM EXISTING SULFURIC ACID PLANTS

62.6875 Identification plan—negative declaration.

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TOTAL REDUCED SULFUR EMISSIONS FROM EXISTING KRAFT PULP MILLS

- 62.6880 Identification of plan—negative declaration.

FLUORIDE EMISSIONS FROM EXISTING PRIMARY ALUMINUM REDUCTION PLANTS

- 62.6910 Identification of plan—negative declaration.

Subpart DD [Reserved]

Subpart EE—New Hampshire

FLUORIDE EMISSIONS FROM PHOSPHATE FERTILIZER PLANTS

- 62.7350 Identification of plan—negative declaration.

SULFURIC ACID MIST EMISSIONS FROM SULFURIC ACID PRODUCTION UNITS

- 62.7375 Identification of plan—negative declaration.

FLUORIDE EMISSIONS FROM EXISTING PRIMARY ALUMINUM PLANTS

- 62.7400 Identification of sources—negative declaration.

Subpart FF—New Jersey

FLUORIDE EMISSIONS FROM PHOSPHATE FERTILIZER PLANTS

- 62.7600 Identification of plan—negative declaration.

TOTAL REDUCED SULFUR EMISSIONS FROM KRAFT PULP MILLS

- 62.7601 Identification of plan—negative declaration.

FLUORIDE EMISSIONS FROM PRIMARY ALUMINUM REDUCTION PLANTS

- 62.7602 Identification of plan—negative declaration.

Subpart GG—New Mexico

- 62.7850 Identification of plan.

SULFURIC ACID MIST EMISSIONS FROM SULFURIC ACID PLANTS

- 62.7851 Identification of sources.

FLUORIDE EMISSIONS FROM PRIMARY ALUMINUM PLANTS

- 62.7852 Identification of plan—negative declaration.

TOTAL REDUCED SULFUR EMISSIONS FROM KRAFT PULP MILLS

- 62.7853 Identification of plan—negative declaration.

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FLUORIDE EMISSIONS FROM PHOSPHATE FERTILIZER PLANTS

- 62.7854 Identification of plan—negative declaration.

Subpart HH—New York

FLUORIDE EMISSIONS FROM PHOSPHATE FERTILIZER PLANTS

- 62.8100 Identification of plan—negative declaration.

SULFURIC ACID MIST EMISSIONS FROM EXISTING SULFURIC ACID PLANTS

- 62.8102 Identification of plan.

FLUORIDE EMISSIONS FROM EXISTING PRIMARY ALUMINUM PLANTS [RESERVED]

Subpart II—North Carolina

PLAN FOR THE CONTROL OF DESIGNATED POLLUTANTS FROM EXISTING FACILITIES (SECTION 111(d) PLAN)

- 62.8350 Identification of plan.

SULFURIC ACID MIST FROM EXISTING SULFURIC ACID PLANTS

- 62.8351 Identification of sources.

FLUORIDE EMISSIONS FROM EXISTING PRIMARY ALUMINUM PLANTS

- 62.8352 Identification of sources.

TOTAL REDUCED SULFUR EMISSIONS FROM KRAFT PULP MILLS

- 62.8353 Identification of sources.

Subpart JJ—[Reserved]

Subpart KK—Ohio

FLUORIDE EMISSIONS FROM PHOSPHATE FERTILIZER PLANTS

- 62.8850 Identification of plan—negative declaration.

TOTAL REDUCED SULFUR EMISSIONS FROM KRAFT PULP MILLS

- 62.8860 Identification of Plan.—Disapproval.

Subpart LL—Oklahoma

PLAN FOR THE CONTROL OF DESIGNATED POLLUTANTS FROM EXISTING FACILITIES (SECTION 111(d) PLAN)

- 62.9100 Identification of plan.

SULFURIC ACID MIST FROM EXISTING SULFURIC ACID PLANTS

- 62.9110 Identification of sources.

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FLUORIDE EMISSIONS FROM PHOSPHATE FERTILIZER PLANTS

62.9120 Identification of plan—negative declaration.

FLUORIDE EMISSIONS FROM PRIMARY ALUMINUM PLANTS

62.9130 Identification of plan—negative declaration.

TOTAL REDUCED SULFUR FROM EXISTING KRAFT PULP MILLS

62.9140 Identification of source.

Subpart MM—Oregon

PLAN FOR THE CONTROL OF DESIGNATED POL- LUTANTS FROM EXISTING FACILITIES (SEC- TION 111(d) PLAN)

62.9350 Identification of plan.

FLUORIDE EMISSIONS FROM EXISTING PRIMARY ALUMINUM PLANTS

62.9360 Identification of sources.

FLUORIDE EMISSIONS FROM PHOSPHATE FERTILIZER PLANTS

62.9500 Identification of sources.

SULFURIC ACID MIST EMISSIONS FROM SULFURIC ACID PRODUCTION UNITS

62.9501 Identification of sources.

Subpart NN—Pennsylvania

FLUORIDE EMISSIONS FROM PHOSPHATE FERTILIZER PLANTS

62.9600 Identification of plan—negative declaration.

SULFURIC ACID MIST EMISSIONS FROM EXISTING SULFURIC ACID PLANTS

62.9601 Identification of Plan.

TOTAL REDUCED SULFUR EMISSIONS FROM EXISTING KRAFT PULP MILLS

62.9610 Identification of plan—negative declaration.

FLUORIDE EMISSIONS FROM EXISTING PRIMARY ALUMINUM PLANTS

62.9620 Identification of plan—negative declaration.

Subpart OO—Rhode Island

TOTAL REDUCED SULFUR EMISSIONS FROM EXISTING KRAFT PULP MILLS

62.9850 Identification of plan—negative declaration.

SULFURIC ACID MIST EMISSIONS FROM SULFURIC ACID PRODUCTION UNITS

62.9875 Identification of plan—negative declaration.

TOTAL REDUCED SULFUR EMISSIONS FROM EXISTING KRAFT PULP MILLS

62.9900 Identification of plan—negative declaration.

FLUORIDE EMISSIONS FROM EXISTING PRIMARY ALUMINUM PLANTS

62.9950 Identification of plan—negative declaration.

Subpart PP—South Carolina

PLAN FOR THE CONTROL OF DESIGNATED POL- LUTANTS FROM EXISTING FACILITIES (SEC- TION 111(d) PLAN)

62.10100 Identification of plan.

SULFURIC ACID MIST FROM EXISTING SULFURIC ACID PLANTS

62.10110 Identification of sources.

TOTAL REDUCED SULFUR EMISSIONS FROM KRAFT PULP MILLS

62.10120 Identification of sources.

FLUORIDE EMISSIONS FROM PHOSPHATE FERTILIZER PLANTS

62.10130 Identification of plan—negative declaration.

FLUORIDE EMISSIONS FROM EXISTING PRIMARY ALUMINUM REDUCTION PLANTS

62.10140 Identification of plan—negative declaration.

Subparts QQ—[Reserved]

Subparts RR—Tennessee

FLUORIDE EMISSIONS FROM PHOSPHATE FERTILIZER PLANTS

62.10602 Identification of sources—negative declaration.

Subpart SS—[Reserved]

Subpart TT—Utah

FLUORIDE FROM EXISTING PHOSPHATE FERTILIZER PLANTS

62.11100 Identification of plan.

Subpart UU—Vermont

FLUORIDE EMISSIONS FROM PHOSPHATE FERTILIZER PLANTS

62.11350 Identification of plan—negative declaration.

§ 62.01

SULFURIC ACID MIST EMISSIONS FROM SULFURIC ACID PRODUCTION UNITS

- 62.11375 Identification of plan—negative declaration.

TOTAL REDUCED SULFUR EMISSIONS FROM EXISTING KRAFT PULP MILLS

- 62.11400 Identification of plan—negative declaration.

FLUORIDE EMISSIONS FROM EXISTING PRIMARY ALUMINUM PLANTS

- 62.11425 Identification of plan—negative declaration.

Subpart VV—Virginia

FLUORIDE EMISSIONS FROM PHOSPHATE FERTILIZER PLANTS

- 62.11600 Identification of plan—negative declaration.

SULFURIC ACID MIST EMISSIONS FROM EXISTING SULFURIC ACID PLANTS

- 62.11601 Identification of plan.
62.11602 Emission standards and compliance schedules.

TOTAL REDUCED SULFUR EMISSIONS FROM EXISTING KRAFT PULP MILLS

- 62.11610—62.11619 [Reserved—plan not submitted]

FLUORIDE EMISSIONS FROM EXISTING PRIMARY ALUMINUM PLANTS

- 62.11620 Identification of plan—negative declaration.

Subpart WW—Washington

FLUORIDE EMISSIONS FROM PHOSPHATE FERTILIZER PLANTS

- 62.11850 Identification of plan—negative declaration.

Subpart XX—West Virginia

FLUORIDE EMISSIONS FROM PHOSPHATE FERTILIZER PLANTS

- 62.12100 Identification of plan—negative declaration.

Subpart YY—Wisconsin

FLUORIDE EMISSIONS FROM PHOSPHATE FERTILIZER PLANTS

- 62.12350 Identification of plan—negative declaration.

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Subparts ZZ—AAA—[Reserved]

Subpart BBB—Puerto Rico

FLUORIDE EMISSIONS FROM PHOSPHATE FERTILIZER PLANTS

- 62.13100 Identification of plan—negative declaration.

SULFURIC ACID MIST EMISSIONS FROM SULFURIC ACID PRODUCTION PLANTS

- 62.13101 Identification of plan—negative declaration.

FLUORIDE EMISSIONS FROM PRIMARY ALUMINUM REDUCTION PLANTS

- 62.13102 Identification of plan—negative declaration.

TOTAL REDUCED SULFUR FROM KRAFT PULP MILLS

- 62.13103 Identification of plan—negative declaration.

Subpart CCC—Virgin Islands

FLUORIDE EMISSIONS FROM PHOSPHATE FERTILIZER PLANTS

- 62.13350 Identification of plan—negative declaration.

SULFURIC ACID MIST EMISSIONS FROM SULFURIC ACID PRODUCTION PLANTS

- 62.13351 Identification of plan—negative declaration.

TOTAL REDUCED SULFUR EMISSIONS FROM KRAFT PULP MILLS

- 62.13352 Identification of plan—negative declaration.

FLUORIDE EMISSIONS FROM PRIMARY ALUMINUM REDUCTION PLANTS

- 62.13353 Identification of plan—negative declaration.

AUTHORITY: Secs. 111 and 301(a), Clean Air Act, as amended (42 U.S.C. 7413 and 7601), unless otherwise noted.

SOURCE: 43 FR 51393, Nov. 3, 1978, unless otherwise noted.

Subpart A—General Provisions

§ 62.01 Definitions.

As used in this part, all terms not defined herein shall have the meaning given to them in the Clean Air Act and in Part 60 of this chapter.

§ 62.02 Introduction.

(a) This part sets forth the Administrator's approval and disapproval of State plans for control of designated pollutants and facilities, and the Administrator's promulgation of such plans or portions thereof. Approval of a plan or any portion of a plan is based on a determination by the Administrator that it meets the requirements of section 111(d) of the act and the provisions of Part 60 of this chapter.

(b) If a State does not submit a complete, approvable plan, the Administrator may then promulgate a substitute plan or part of a plan. The promulgated provision, plus the approved parts of the State plan, constitute the applicable plan for purposes of the act.

(c) The Administrator will promulgate substitute provisions for the disapproved regulatory provisions only. If a nonregulatory provision is disapproved, however, it will be noted in this part and a detailed explanation will be sent to the State.

(d) All approved regulatory provisions of each plan are incorporated by reference in this part. Section 62.12 provides information on availability of applicable plans. The Administrator and State and local agencies shall enforce (1) regulatory provisions of a plan approved or promulgated by the Administrator, and (2) all permit conditions or denials issued in carrying out the approved or promulgated regulations for the review of designated facilities.

(e) Each State's plan is dealt with in a separate subpart, with separate headings for different pollutants and facilities. The plans shall include an introductory section identifying the plan by name and the date of its submittal. Additional sections are included as necessary to specifically identify disapproved provisions, to set forth reasons for disapproval, and to set forth provisions of the plan promulgated by the Administrator. Except as otherwise specified, all supplemental information submitted to the Administrator with respect to any plan has been submitted by the Governor of the State.

(f) Revisions to applicable plans will be included in this part when approved or promulgated by the Administrator.

§ 62.03 Extensions.

The Administrator may, whenever he determines necessary, extend the period for submission of any plan or plan revision or portion thereof.

§ 62.04 Approval status.

The approval status of each State's plan or portions thereof, are set forth in each subpart. All plans are approved unless specifically disapproved in the appropriate subpart.

§ 62.05 Legal authority.

(a) The Administrator's determination of the absence or inadequacy of legal authority required to be included in the plan is set forth in each subpart. This includes the legal authority of local agencies and State governmental agencies other than an air pollution control agency if such other agencies are assigned responsibility for carrying out a plan or portion thereof.

(b) No legal authority as such is promulgated by the Administrator. Where required regulatory provisions are not included in the plan by the State because of inadequate authority, substitute provisions are promulgated by the Administrator.

§ 62.06 Negative declarations.

A State may submit to the Administrator a letter certifying that no designated facilities exist in the State if such is the case. The negative declaration will be in lieu of a plan.

§ 62.07 Emission standards, compliance schedules.

(a) In each subpart, emission standards and compliance schedules which have been disapproved by the Administrator are identified, and those promulgated by the Administrator are set forth.

(b) The Administrator's approval or promulgation of any compliance schedule shall not affect the responsibility of the owner or operator to comply with any applicable emission limitation on or after the date for

final compliance specified in the applicable schedule.

§ 62.08 Emission inventories and source surveillance.

(a) Each subpart identifies the plan provisions for source surveillance which are disapproved, and sets forth the Administrator's promulgation of necessary provisions for requiring designated sources to maintain records, make reports, and submit information.

(b) The Administrator will not promulgate provisions for disapproved State or local agency procedures for testing, inspection, investigation, or detection. However, detailed critiques of such portions will be provided to the State.

§ 62.09 Revision of plans by Administrator.

After notice and opportunity for public hearing in each affected State, the Administrator may revise any provision of an applicable plan if:

(a) The provision was promulgated by the Administrator and

(b) The plan, as revised, will be consistent with the Act and with the requirements of Part 60, Subpart B of this chapter.

§ 62.10 Submission to Administrator.

Except as otherwise provided in § 60.23 of this chapter, all requests, reports, applications, submittals, and other communications to the Administrator pursuant to this part shall be submitted in duplicate and addressed to the appropriate Regional Office of the Environmental Protection Agency, to the attention of the Director, Air and Hazardous Materials Division (Environmental Programs Division in Region II). The Regional Offices are as follows:

Region and jurisdiction covered	Address
I—Connecticut, Maine, Massachusetts, New Hampshire, Rhode Island, Vermont.	JFK Federal Building, Boston, Mass. 02203.
II—New York, New Jersey, Puerto Rico, Virgin Islands.	Federal Office Building, 26 Federal Plaza, New York, N.Y. 10007. Curtis Building, 8th and Walnut Sts., Philadelphia, Pa. 19106.
III—Delaware, District of Columbia, Pennsylvania, Maryland, Virginia, West Virginia.	

Region and jurisdiction covered	Address
IV—Alabama, Florida, Georgia, Mississippi, Kentucky, North Carolina, South Carolina, Tennessee.	345 Courtland NE, Atlanta, Ga. 30308.
V—Illinois, Indiana, Michigan, Minnesota, Ohio, Wisconsin.	Federal Building, 2 South Dearborn Chicago, Ill. 60606.
VI—Arkansas, Louisiana, New Mexico, Oklahoma, Texas.	1st International Bldg., 1201 Elm St., Dallas, Tex. 75270.
VII—Iowa, Kansas, Missouri, Nebraska.	1735 Baltimore St., Kansas City, Mo. 64108.
VIII—Colorado, Montana, North Dakota, South Dakota, Utah, Wyoming.	916 Lincoln Towers, 1860 Lincoln St., Denver, Colo. 80202.
IX—Arizona, California, Hawaii, Nevada, Guam, American Samoa.	215 Fremont St., San Francisco, Calif. 94101.
X—Washington, Oregon, Idaho, Alaska.	1200 6th Ave., Seattle, Wash. 98101.

§ 62.11 Severability.

The provisions promulgated in this part and the various applications thereof are distinct and severable. If any provision of this part or the application thereof to any person or circumstances is held invalid, such invalidity shall not affect other provisions or application of such provision to other persons or circumstances which can be given effect without the invalid provision or application.

§ 62.12 Availability of applicable plans.

Copies of the applicable plans will be available for public inspection at the following locations:

(a) The offices of the Directors, Air and Hazardous Materials Divisions at EPA Regional Offices I, III-X, and the Director, Environmental Programs Division at EPA Regional Office II. The addresses and jurisdictions covered by these appear in § 62.10.

(b) Public Information Reference Unit, Library Systems Branch, EPA (PM 213), 401 M Street SW., Washington, D.C. 20460.

Subpart B—Alabama

AUTHORITY: Sec. 110(a) and 111(d), Clean Air Act (42 U.S.C. 7410(a) and 7411(d)).

SOURCE: 48 FR 31402, July 8, 1983, unless otherwise noted.

PLAN FOR THE CONTROL OF DESIGNATED POLLUTANTS FROM EXISTING FACILITIES (SECTION 111(d) PLAN)

§ 62.100 Identification of plan.

(a) *Identification of plan.* Alabama Designated Facility Plan (Section 111(d) Plan).

(b) *The plan was officially submitted as follows.* (1) Control of sulfuric acid mist emissions from existing sulfuric acid production units, submitted on May 18, 1980;

(2) Control of fluoride emissions from existing phosphate fertilizer plants, submitted on April 10, 1978.

(c) *Designated facilities.* The plan applies to existing facilities in the following categories of sources:

- (1) Sulfuric acid plants;
- (2) Phosphate fertilizer plants.

SULFURIC ACID MIST FROM EXISTING SULFURIC ACID PLANTS

§ 62.101 Identification of sources.

The plan applies to existing facilities at the following sulfuric acid plants:

- (a) Acid plants operated by
 - (1) Reichhold Chemical Company in Tuscaloosa,
 - (2) Stauffer Chemical Company in Mobile, and
 - (3) Estech Chemical in Dothan.
- (b) There are no oleum plants.
- (c) There are not sulfur-burning plants.
- (d) There are no bound sulfur feed-stock plants.

FLUORIDE EMISSIONS FROM PHOSPHATE FERTILIZER PLANTS

§ 62.102 Identification of sources.

The plan currently does not identify any sources subject to its fluoride emission limits.

Subpart C—Alaska

SOURCE: 44 FR 76281, Dec. 26, 1979, unless otherwise noted.

FLUORIDE EMISSIONS FROM PHOSPHATE FERTILIZER PLANTS

§ 62.350 Identification of plan—negative declaration.

The Alaska Department of Environmental Conservation submitted on

June 9, 1977, certification that there are no existing phosphate fertilizer plants in the State subject to Part 60, Subpart B of this chapter.

ACID MIST FROM SULFURIC ACID PLANTS

§ 62.351 Identification of plan—negative declaration.

The Alaska Department of Environmental Conservation submitted on June 9, 1977, certification that there are no existing sulfuric acid plants in the State subject to Part 60, Subpart B of this chapter.

TOTAL REDUCED SULFUR EMISSIONS FROM KRAFT PULP MILLS

§ 62.352 Identification of plan—negative declaration.

The Alaska Department of Environmental Conservation submitted on June 9, 1977, certification that there are no existing kraft pulp mills in the State subject to Part 60, Subpart B of this chapter.

FLUORIDE EMISSIONS FROM PRIMARY ALUMINUM REDUCTION PLANTS

§ 62.353 Identification of plan—negative declaration.

The Alaska Department of Environmental Conservation submitted on June 9, 1977, certification that there are no existing primary aluminum reduction plants in the State subject to Part 60, Subpart B of this chapter.

Subpart D—[Reserved]

Subpart E—Arkansas

SOURCE: 47 FR 20491, May 12, 1982, unless otherwise noted.

PLAN FOR THE CONTROL OF DESIGNATED POLLUTANTS FROM EXISTING FACILITIES (SECTION 111(d) PLAN)

§ 62.850 Identification of plan.

(a) *Identification of plan:* Arkansas Plan for the Control of Designated Pollutants from Existing Plants (Section 111(d) Plan).

(b) *The plan was officially submitted as follows:*

(1) Control of sulfuric acid mist from sulfuric acid plants, and fluoride emissions from phosphate fertilizer plants, submitted on July 11, 1979, having been adopted by the State on May 25, 1979, and letter dated August 6, 1981.

(2) Control of total reduced sulfur (TRS) emissions from existing kraft pulp mills submitted by the Governor on February 28, 1983, and adopted by the State on January 28, 1983.

(c) Designated facilities: The plan applies to existing facilities in the following categories of sources:

- (1) Sulfuric acid plants.
- (2) Phosphate fertilizer plants.
- (3) Kraft pulp mills.

[47 FR 20491, May 12, 1982, as amended at 49 FR 35773, Sept. 12, 1984]

§ 62.852 Emission inventories, source surveillance, reports.

(a) For purposes of this subpart, the terms "the applicable plan" and "the plan" in 40 CFR 52.178(b), which provides for public availability of emission data, include this plan.

FLUORIDE EMISSIONS FROM EXISTING PHOSPHATE FERTILIZER PLANTS

§ 62.854 Identification of sources.

The plan applies to existing facilities at the following existing phosphate fertilizer plant:

- (1) Allied Chemical Company at Helena, Arkansas.

SULFURIC ACID MIST EMISSIONS FROM EXISTING SULFURIC ACID PLANTS

§ 62.855 Identification of sources.

(a) The plan applies to existing facilities at the following existing sulfuric acid plants:

- (1) Olin Corporation in North Little Rock, Arkansas.
- (2) The Monsanto Company in Eldorado, Arkansas.

TOTAL REDUCED SULFUR EMISSIONS FROM EXISTING KRAFT PULP MILLS

§ 62.865 Identification of sources.

(a) The plan applies to existing facilities at the following kraft pulp mill plants:

- (1) International Paper Company in Camden, Arkansas.

(2) International Paper Company in Pine Bluff, Arkansas.

(3) Arkansas Kraft Corporation in Morrilton, Arkansas.

(4) Weyerhaeuser Company in Pine Bluff, Arkansas.

(5) Georgia-Pacific Corporation in Crossett, Arkansas.

(6) Wekoosa Paper Company in Ardmore, Arkansas.

(7) Potlatch Corporation of McGehee, Arkansas.

[49 FR 35773, Sept. 12, 1984]

§ 62.866 Compliance schedule.

The Compliance Schedules were submitted on December 16, 1985, by the Governor to control total reduced sulfur emissions from the seven kraft pulp mills identified in § 62.865(a). The schedules specify final compliance dates and enforceable increments to be as expeditiously as practicable but not more than six years from approval of the state regulations; i.e., October 12, 1990.

[51 FR 40803, Nov. 10, 1986]

Subpart F—Plan for the Control of Designated Pollutants From Existing Facilities (Section 111(d) Plan)

AUTHORITY: Sec. 111 of the Clean Air Act as amended (42 U.S.C. 7411).

SOURCE: 47 FR 28100, June 29, 1982, unless otherwise noted.

§ 62.1100 Identification of plan.

(a) State of California Designated Facility Plan (Section 111(d) Plan).

(b) The plan was officially submitted as follows:

(1) Control of fluoride emissions from existing facilities at phosphate fertilizer plants, submitted on February 26 and July 16, 1979 and April 1, 1980 having been adopted by the Districts on December 1 and 6, 1979 and January 9, 1979. A letter clarifying the plan was submitted on March 27, 1979. Revisions to the plan were submitted on September 23, 1980 and February 1 and July 6, 1981.

(2) Control of sulfuric acid mist from existing facilities at sulfuric acid production units, submitted on February

26, July 16, and September 7, 1979 and April 7, 1980, having been adopted by the Districts on December 1 and 6, 1978 and January 9, 1979. Revisions to the plan were submitted on October 31, 1980, February 18, and May 1, 1981.

(3) Control of total reduced sulfur (TRS) emissions from existing kraft pulping mills submitted as follows:

(i) 9-25-79; submittal of existing rules; (a) Bay Area Air Quality Management District (AQMD) Rule 1, Regulation 12—Kraft Pulp Mills.

(b) Humboldt County Air Pollution Control District Regulation 1; Rule 130—Definitions, Rule 240—Permit to Operate, Rule 450—Sulfide Emissions from Kraft Pulp Mills.

(c) Shasta County Air Pollution Control District Rule 3:2—Specific Air Contaminants.

(ii) 3-21-80; Clarification of Bay Area Rule 1, Regulation 12—Kraft Pulp Mills.

(iii) 4-7-80; Summary of district rules and State laws that meet the requirements of 40 CFR, Parts 60.23-60.26 for Designated Facilities in general.

(iv) 5-29-80; revision of Bay Area AQMD Rule 1, Regulation 12—Kraft Pulp Mills.

(v) 9-5-80; Evidence of public hearing and annual report schedule defined for Bay Area Rule 1, Regulation 12—Kraft Pulp Mills.

(vi) 11-4-81; (a) Humboldt County APCD Rules 130—Definitions; 240—Permit to Operate; and 450—Kraft Pulp Mills amended (7-28-81).

(b) Shasta County APCD Rule 3:2—Specific Contaminants amended (8-4-81).

(c) A summary of compliance of all districts with the requirements set forth in 40 CFR 60.23 through 60.26.

(d) A list of witnesses appearing at Humboldt and Shasta Counties public hearings and a summary of testimonies Statewide emissions inventory of all TRS sources in the State.

(e) Designated facilities: The plans apply to existing facilities in the following categories of sources:

(1) Existing phosphate fertilizer plants.

(2) Existing sulfuric acid production units.

(3) Existing Kraft pulp mills.

[47 FR 28100, June 29, 1982, as amended at 47 FR 47384, Oct. 26, 1982]

FLUORIDE EMISSIONS FROM EXISTING PHOSPHATE FERTILIZER PLANTS

§ 62.1101 Identification of sources.

The plan applies to existing facilities at the following phosphate fertilizer plants:

(a) Occidental Chemical Company in San Joaquin County.

(b) Simplot Company in Kings County.

(c) Valley Nitrogen Products, Inc., in Fresno County.

SULFURIC ACID MIST EMISSIONS FROM EXISTING SULFURIC ACID PRODUCTION UNITS

§ 62.1102 Identification of sources.

The plan applies to existing facilities at the following sulfuric acid production units:

(a) Allied Chemical Corporation in Alameda County.

(b) Monsanto Company in Alameda County.

(c) Occidental Chemical Company in Fresno County.

(d) Stauffer Chemical Company in Alameda County.

(e) Valley Nitrogen Products, Inc. in Kern County.

FLUORIDE EMISSIONS FROM PRIMARY ALUMINUM REDUCTION PLANTS

§ 62.1103 Identification of plan—negative declaration.

TOTAL REDUCED SULPHUR EMISSIONS FROM EXISTING KRAFT PULP MILLS

§ 62.1104 Identification of sources.

The plan applies to existing facilities at the following Kraft pulp mills:

(a) Louisiana Pacific, Antioch, Contra Costa County Pulp Mill.

(b) Louisiana Pacific Corp., Samoa Complex.

(c) Crown Simpson Pulp Company, Fairhaven.

(d) Simpson Paper Company, Shasta County Pulp Mill.

[47 FR 47385, Oct. 26, 1982]

Subpart G—[Reserved]

Subpart H—Connecticut

FLUORIDE EMISSIONS FROM PHOSPHATE FERTILIZER PLANTS

§ 62.1600 Identification of plan—negative declaration.

The State Department of Environmental Protection submitted on November 30, 1977, a letter certifying that there are no existing phosphate fertilizer plants in the state subject to Part 60, Subpart B of this chapter.

[44 FR 54052, Sept. 18, 1979]

SULFURIC ACID MIST EMISSIONS FROM SULFURIC ACID PRODUCTION UNITS

§ 62.1625 Identification of plan—negative declaration.

The State Department of Environmental Protection submitted on November 30, 1977, a letter certifying that there are no existing sulfuric acid plants in the state subject to Part 60, Subpart B of this chapter.

[44 FR 54052, Sept. 18, 1979]

TOTAL REDUCED SULFUR EMISSIONS FROM EXISTING KRAFT PULP MILLS

§ 62.1650 Identification of plan—negative declaration.

The State Department of Environmental Protection submitted on December 28, 1988, a letter certifying that there are no existing kraft pulp mills in the State subject to Part 60, Subpart B of this chapter.

[54 FR 9046 Mar. 3, 1989]

FLUORIDE EMISSIONS FROM EXISTING PRIMARY ALUMINUM PLANTS

§ 62.1700 Identification of plan—negative declaration.

The State Department of Environmental Protection submitted on December 28, 1988, a letter certifying that there are no existing primary aluminum reduction plants in the State subject to Part 60, Subpart B of this chapter.

[54 FR 9046 Mar. 3, 1989]

Subpart I—Delaware

FLUORIDE EMISSIONS FROM PHOSPHATE FERTILIZER PLANTS

§ 62.1850 Identification of plan—negative declaration.

The Delaware Department of Natural Resources and Environmental Control submitted on November 7, 1977, a letter certifying that there are no existing phosphate fertilizer plants in the State subject to Part 60, Subpart B of this chapter.

[45 FR 43412, June 27, 1980]

SULFURIC ACID MIST FROM EXISTING SULFURIC ACID PLANTS

§ 62.1875 Identification of plan.

(a) Title of plan: State implementation plan for control of sulfuric acid mist from existing sulfuric acid plants.

(b) The plan was officially submitted on September 8, 1978 with amendments submitted on December 28, 1980.

(c) Identification of Sources: The plan includes the following sulfuric acid plants:

(1) Allied Chemical Company, Clarks Summit (New Castle County).

[47 FR 10536, Mar. 11, 1982]

TOTAL REDUCED SULFUR EMISSIONS FROM KRAFT PULP MILLS

§ 62.1900 Identification of plan—negative declaration.

The Delaware Department of Natural Resources and Environmental Control submitted on September 8, 1982, a letter certifying that there are no kraft pulp mills in the State subject to Part 60, Subpart B of this chapter.

[48 FR 10652, Mar. 14, 1983]

FLUORIDE EMISSIONS FROM PRIMARY ALUMINUM REDUCTION PLANTS

§ 62.1925 Identification of plan—negative declaration.

The Delaware Department of Natural Resources and Environmental Control submitted on September 8, 1982, a letter certifying that there are no primary aluminum reduction plants in the State subject to Part 60, Subpart B of this chapter.

the State subject to Part 60, Subpart B of this chapter.

[48 FR 10652, Mar. 14, 1983]

Subpart J—District of Columbia

FLUORIDE EMISSIONS FROM PHOSPHATE FERTILIZER PLANTS

§ 62.2100 Identification of plan—negative declaration.

The Department of Environmental Services submitted on December 12, 1977 a letter certifying that there are no existing phosphate fertilizer plants in the District subject to Part 60, Subpart B of this chapter.

[45 FR 43412, June 27, 1980]

SULFURIC ACID MIST EMISSIONS FROM EXISTING SULFURIC ACID PLANTS

§ 62.2101 Identification of plan—negative declaration.

The Director, Department of Environmental Services submitted on March 7, 1978 a letter certifying there are no existing sulfuric acid production units in the District subject to Part 60, Subpart B of this chapter.

[46 FR 41783, Aug. 18, 1981]

TOTAL REDUCED SULFUR EMISSIONS FROM EXISTING KRAFT PULP MILLS

§ 62.2110 Identification of plan—negative declaration.

The Mayor of the District of Columbia submitted on July 16, 1980 a letter certifying there are no existing primary kraft pulp mills in the District subject to Part 60, Subpart B of this chapter.

[46 FR 41783, Aug. 18, 1981]

FLUORIDE EMISSIONS FROM EXISTING PRIMARY ALUMINUM PLANTS

§ 62.2120 Identification of plan—negative declaration.

The Mayor of the District of Columbia submitted on May 29, 1980 a letter certifying there are no existing primary aluminum plants in the District subject to Part 60, Subpart B of this chapter.

[46 FR 41783, Aug. 18, 1981]

Subpart K—Florida

AUTHORITY: Secs. 110(a) and 111(d), Clean Air Act (42 U.S.C. 7410(a) and 7411(d)).

SOURCE: 48 FR 31402, July 8, 1983, unless otherwise noted.

PLAN FOR THE CONTROL OF DESIGNATED POLLUTANTS FROM EXISTING FACILITIES (SECTION 111(d) PLAN)

§ 62.2350 Identification of plan.

(a) *Identification of plan.* Florida Designated Facility Plan (Section 111(d) Plan).

(b) *The plan was officially submitted as follows.* (1) Control of sulfuric acid mist emissions from existing sulfuric acid production units, submitted on December 14, 1978.

(2) Control of total reduced sulfur (TRS) emissions from existing kraft pulp mills and tall oil plants (both new and existing) submitted on May 24, 1985, and revision submitted on June 10, 1986, by the Florida Department of Environmental Regulation (FDER). No action is taken on sections 17-2.600(4)(c)7 and 8.

(3) The final compliance date to achieve the TRS emission limits for the black liquor evaporation system, the batch digester system and the continuous digester system for St. Joe Paper Company in Port St. Joe is September 14, 1989.

(4) The final compliance date to achieve TRS emission limits for the No. 5 Multiple Effect Evaporation System, batch digester system and Kamyr digester system for Container Corporation of America in Fernadina Beach, Florida is June 1, 1990.

(c) *Designated facilities.* The plan applies to existing facilities in the following categories of sources:

- (1) Sulfuric acid plants.
- (2) Kraft pulp mills.

[48 FR 31402, July 8, 1983, as amended at 53 FR 30053, Aug. 10, 1988; 54 FR 40003, Sept. 29, 1989; 54 FR 48102, Nov. 21, 1989]

SULFURIC ACID MIST FROM EXISTING SULFURIC ACID PLANTS

§ 62.2351 Identification of sources.

The plan applies to existing facilities at the following sulfuric acid plants:

- (a) Acid plants operated by:

- (1) Occidental Petroleum Company in Hamilton County,
- (2) AMAX Phosphate Inc. in Manatee County,
- (3) Conserv Chemical in Nichols County,
- (4) Farmland Industry in Bartow County,
- (5) W. R. Grace Company in Polk County,
- (6) Royster Fertilizer in Polk County,
- (7) USS Agrichemicals in Polk County,
- (8) Central Farmers Co-Op in Polk County,
- (9) Agrico Chemical Company in Polk County,
- (10) Gardinier, Inc. in Hillsborough County, and
- (11) ESTECH in Polk County.
- (b) There are no oleum plants.
- (c) There are no sulfur-burning plants.
- (d) There are no bound sulfur feed-stock plants.

FLUORIDE EMISSIONS FROM PRIMARY ALUMINUM REDUCTION PLANTS

§ 62.2352 Identification of source—Negative declaration.

The Florida Department of Environmental Regulation submitted on April 22, 1985, a letter certifying that there are no existing primary aluminum reduction plants in the State subject to Part 60, Subpart B of this chapter.

[50 FR 26204, June 25, 1985]

TOTAL REDUCED SULFUR EMISSIONS FROM KRAFT PULP MILLS AND TALL OIL PLANTS

§ 62.2353 Identification of sources.

The plan applies to existing facilities at the following existing kraft pulp plants and tall oil plants:

- (a) Alton Packaging Corporation in Jacksonville
- (b) Buckeye Cellulose Corporation in Perry
- (c) Champion International Corporation (Formerly St. Regis Paper Company) in Cantonment
- (d) Container Corporation of America in Fernandina Beach
- (e) Georgia-Pacific Corporation in Palatka

- (f) Jacksonville Kraft Paper Company in Jacksonville
- (g) St. Joe Paper Company in Port St. Joe
- (h) Southwest Forest Industries in Panama City
- (i) Arizona Chemical Company (Tall Oil Plant) in Panama City
- (j) Sylvachem Corporation (Tall Oil Plant) in Port St. Joe

[53 FR 30053, Aug. 10, 1988]

§ 62.2354 Compliance schedules.

The State of Florida has provided that the individual source compliance schedules would be developed and submitted by the affected sources to the State following plan adoption; and that the increments of progress pursuant to 40 CFR 60.21(h) would be specified at that time; this is an acceptable procedure pursuant to 40 CFR 60.24(e)(2). However, the State must submit these schedules to EPA for approval; and these schedules must meet the public hearing requirements of 40 CFR 60.23 or ones deemed equivalent by the Administrator pursuant to 40 CFR 60.23(g).

[53 FR 30053, Aug. 10, 1988]

Subpart L—Georgia

AUTHORITY: Secs. 110(a) and 111(d), Clean Air Act (42 U.S.C. 7410(a) and 7411(d)).

SOURCE: 48 FR 31402, July 8, 1983, unless otherwise noted.

PLAN FOR THE CONTROL OF DESIGNATED POLLUTANTS FROM EXISTING FACILITIES (SECTION 111(d) PLAN)

§ 62.2600 Identification of plan.

(a) *Identification of plan.* Georgia Designated Facility Plan (Section 111(d) Plan).

(b) *The plan was officially submitted as follows.* (1) Control of sulfuric acid mist emissions from existing sulfuric acid production units, submitted on January 31, 1978;

(2) Control of total reduced sulfur emissions from existing facilities at Kraft pulp mills, submitted on January 8, 1982.

(3) A compliance schedule for sources subject to the plan for the control of total reduced sulfur emissions.

sions from existing kraft pulp mills and a starting date for such rule, submitted on June 3, 1988.

(c) *Designated facilities.* The plan applies to existing facilities in the following categories of sources:

- (1) Sulfuric acid plants;
- (2) Kraft pulp mills.

**SULFURIC ACID MIST FROM EXISTING
SULFURIC ACID PLANTS**

[48 FR 31402, July 8, 1983, as amended at 53 FR 38291, Sept. 30, 1988]

§ 62.2601 Identification of sources.

The plan applies to existing facilities at the following sulfuric acid plants:

(a) Sulfur-burning plants operated by:

(1) American Cyanamid Company in Savannah, and

(2) Cities Service Company in Augusta.

(b) Oleum plant of Cities Service Company in Augusta.

(c) There are no bound sulfur feed-stock plants.

**FLUORIDE EMISSIONS FROM PHOSPHATE
FERTILIZER PLANTS**

§ 62.2602 Identification of sources—Negative declaration.

The Georgia Environmental Protection Division submitted on July 14, 1977, a letter certifying that there are no existing phosphate fertilizer plants in the State subject to Part 60, Subpart B, of this chapter.

**TOTAL REDUCED SULFUR EMISSIONS
FROM KRAFT PULP MILLS**

§ 62.2603 Identification of sources.

The plan applies to existing facilities at the following Kraft pulp mills:

(a) Continental Can in Augusta.

(b) Continental Can in Port Wentworth.

(c) Brunswick in Brunswick.

(d) Georgia Kraft in Rome.

(e) Georgia Kraft in Macon.

(f) Gilman in St. Marys.

(g) Great Southern in Cedar Springs.

(h) Interstate in Riceboro.

(i) ITT Rayonier in Jesup.

(j) Owens-Illinois in Valdosta, and

(k) Union Camp in Savannah.

§ 62.2604 [Reserved]

§ 62.2605 Identification of sources—Negative declaration.

The Georgia Environmental Protection Division submitted a letter on October 19, 1983, certifying that there are no existing primary aluminum reduction plants in the State of Georgia subject to 40 CFR Part 60, Subpart B, of this chapter.

[49 FR 3855, Jan. 31, 1984]

Subpart M—[Reserved]

Subpart N—Idaho

**FLUORIDE EMISSIONS FROM EXISTING
PRIMARY ALUMINUM PLANTS**

§ 62.3100 Identification of plan—negative declaration.

The State of Idaho Department of Health and Welfare submitted on February 23, 1981, certification that there are no existing primary aluminum plants in the State subject to Part 60, Subpart B of this chapter.

[47 FR 47250, Oct. 25, 1982]

Subpart O—Illinois

**SULFURIC ACID MIST EMISSIONS FROM
EXISTING SULFURIC ACID PRODUCTION PLANTS**

§ 62.3300 Identification of plan.

(a) Title of Plan: "Illinois Plan for the Control of Sulfuric Acid Mist from Existing Contract Process Sulfuric Acid Plants."

(b) The plan was officially submitted on August 10, 1978.

(c) Identification of sources: The plan includes the following sulfuric acid production plants:

(1) Beker Industries in LaSalle County.

(2) U.S.I. Chemical Company in Douglas County.

(3) Mobil Chemical Company in Bureau County.

(4) Swift Chemical Company in Cook County.

(5) American Cyanamid Company in Will County.

(6) Amax Zinc Company in St. Clair County.

(7) Monsanto Company in St. Clair County.

(8) Smith Douglas—Division of Border Chemical in Livingston County.

[46 FR 57896, Nov. 27, 1981]

TOTAL REDUCED SULFUR EMISSIONS FROM KRAFT PULP MILLS

§ 62.3325 Identification of Plan—negative declaration.

The Illinois Environmental Protection Agency submitted on July 23, 1979, a letter certifying that there are no existing kraft pulp mills in the State subject to Part 60, Subpart B of this chapter.

[46 FR 57896 Nov. 27, 1981]

Subpart P—Indiana

FLUORIDE EMISSIONS FROM PHOSPHATE FERTILIZER PLANTS

§ 62.3600 Identification of plan—negative declaration.

The State Board of Health submitted on April 18, 1977, a letter certifying that there are no existing phosphate fertilizer plants in the State subject to Part 60, Subpart B of this chapter.

FLORIDE EMISSIONS FROM EXISTING PRIMARY ALUMINUM PLANTS

§ 62.3625 Identification of plan.

(a) Title of plan: "Fluoride Emission Limitations for Existing Primary Aluminum Plants."

(b) The plan was officially submitted on January 7, 1981 by the Technical Secretary of the Indiana Air Pollution Control Board.

(c) The State on July 17, 1981, submitted Alcoa methods 4075A, 4076A, 913A, 914E and 914F as alternate test methods.

[46 FR 57896, Nov. 27, 1981, as amended at 46 FR 57897, Nov. 27, 1981]

Subpart Q—Iowa

SOURCE: 50 FR 52921, Dec. 27, 1985, unless otherwise noted.

PLAN FOR THE CONTROL OF DESIGNATED POLLUTANTS FROM EXISTING FACILITIES (SECTION 111(d) PLAN)

§ 62.3850 Identification of plan.

(a) Identification of plan: Iowa Plan for Control of Designated Pollutants from Existing Facilities (Section 111(d) Plan).

(b) The plan was officially submitted as follows:

(1) Control of sulfuric acid mist from existing facilities at sulfuric acid production plants, effective on June 12, 1971, having been submitted by the State on February 23, 1978. Additional information was provided in letters of February 7, 1983; May 13, 1985; and June 12, 1985.

(2) Control of fluoride emissions from existing facilities at phosphate fertilizer plants, effective on August 29, 1979, having been submitted by the State on October 19, 1979. Additional information was provided in letters of February 7, 1983; May 13, 1985; and June 12, 1985.

(c) Designated facilities: The plan applies to existing facilities in the following categories of sources:

- (1) Sulfuric acid production plants.
- (2) Phosphate fertilizer plants.

SULFURIC ACID MIST FROM EXISTING SULFURIC ACID PRODUCTION PLANTS

§ 62.3851 Identification of sources.

(a) The plan applies to existing facilities at the following sulfuric acid production plants:

- (1) Agrico Chemical Company, Fort Madison, Iowa
- (2) Koch Sulfur Products Company, Dubuque, Iowa

FLUORIDE EMISSIONS FROM EXISTING PHOSPHATE FERTILIZER PLANTS

§ 62.3852 Identification of sources.

(a) The plan applies to existing facilities at the following phosphate fertilizer plants:

- (1) Agrico Chemical Company, Fort Madison, Iowa.
- (2) Chevron Chemical Company, Fort Madison, Iowa.
- (3) Occidental Chemical Company, Buffalo, Iowa.

Environmental Protection Agency

§ 62.4350

TOTAL REDUCED SULFUR EMISSIONS FROM EXISTING KRAFT PULP MILLS

§ 62.3853 Identification of plan—negative declaration.

Letter from Executive Director of Iowa Department of Environmental Quality submitted on February 7, 1983, certifying that there are no kraft pulp mills in the State of Iowa subject to Part 60, Subpart B of this chapter.

FLUORIDE EMISSIONS FROM EXISTING PRIMARY ALUMINUM REDUCTION PLANTS

§ 62.3854 Identification of plan—negative declaration.

Letter from the Iowa Department of Water, Air and Waste Management submitted on May 13, 1985, certifying that there are no primary aluminum reduction plants in the State of Iowa subject to Part 60, Subpart B of this chapter.

TOTAL REDUCED SULFUR EMISSIONS FROM EXISTING KRAFT PULP MILLS

§ 62.3910 Identification of Plan—Negative Declaration.

Letter from Executive Director of Iowa Department of Environmental Quality submitted on February 7, 1983, certifying that there are no Kraft pulp mills in the State of Iowa, subject to Part 60, Subpart B of this chapter.

[49 FR 43058, Oct. 26, 1984]

Subpart R—Kansas

SOURCE: 49 FR 7234, Feb. 28, 1984, unless otherwise noted.

FLUORIDE EMISSIONS FROM EXISTING PHOSPHATE FERTILIZER PLANTS

§ 62.4100 Identification of plan—negative declaration.

Letter from the Director of the Department of Health and Environment submitted on August 2, 1978, certifying that there are no phosphate fertilizer manufacturing facilities in the State of Kansas.

TOTAL REDUCED SULFUR EMISSIONS FROM EXISTING KRAFT PULP MILLS

§ 62.4125 Identification of plan—negative declaration.

Letter from the Director of the Department of Health and Environment submitted on July 17, 1979, certifying that there are no kraft pulp mills in the State of Kansas.

FLUORIDE EMISSIONS FROM EXISTING PRIMARY ALUMINUM REDUCTION PLANTS

§ 62.4150 Identification of plan—negative declaration.

Letter from the Director, Division of Environment, Kansas Department of Health and Environments submitted on May 23, 1984, certifying that there are no primary aluminum reduction plants on the State of Kansas, subject to Part 60, Subpart B of this chapter.

[49 FR 43058, Oct. 26, 1984]

SULFURIC ACID MIST FROM EXISTING SULFURIC ACID PRODUCTION PLANTS

§ 62.4175 Identification of plan.

(a) *Identification of plan.* State of Kansas Implementation Plan for Control of Sulfuric Acid Mist from Existing Sulfuric Acid Plants.

(b) The Plan was officially submitted on February 6, 1986.

(c) *Identification of sources.* The Plan applies to existing facilities at the following existing sulfuric acid plant: (1) Koch Sulfur Products, DeSoto, Kansas.

[51 FR 37275, Oct. 21, 1986]

Subpart S—Kentucky

SOURCE: 47 FR 22956, May 26, 1982, unless otherwise noted.

PLAN FOR THE CONTROL OF DESIGNATED POLLUTANTS FROM EXISTING FACILITIES (SECTION 111(d) PLAN)

§ 62.4350 Identification of plan.

(a) *Identification of plan:* Kentucky Designated Facility Plan (Section 111(d) Plan).

(b) The plan was officially submitted as follows:

(1) Control of sulfuric acid mist emissions from existing facilities at sulfuric acid plants, total reduced sulfur emissions from existing facilities at kraft pulp mills, fluoride emissions from existing facilities at primary aluminum reduction plants; officially submitted on December 15, 1981.

(c) Designated facilities: The plan applies to existing facilities in the following categories of sources:

- (1) Sulfuric acid plants.
- (2) Kraft pulp mills.
- (3) Primary aluminum reduction plants.

**SULFURIC ACID MIST FROM EXISTING
SULFURIC ACID PLANTS**

§ 62.4351 Identification of sources.

The plan applies to existing facilities at the following sulfuric acid plant: E.I. du Pont sulfuric acid plant in Wurtland, Ky.

**TOTAL REDUCED SULFUR FROM EXISTING
KRAFT PULP MILLS**

§ 62.4352 Identification of sources.

The plan applies to existing facilities at the following kraft pulp mills:

- (a) Westvaco Corp., Fine Papers Division, Wickliffe, Ky.
- (b) Willamette Industries, Corrugated Medium Mill and Bleached Pulp Mill, Hawesville, Kentucky.

**FLUORIDE EMISSIONS FROM EXISTING
PRIMARY ALUMINUM REDUCTION
PLANTS**

§ 62.4353 Identification of sources.

The plan applies to existing facilities at the following primary aluminum reduction plants:

- (a) National Southwire Aluminum, Hawesville, Ky.
- (b) Anaconda Company, Aluminum Division, Henderson, Ky.

**FLUORIDE EMISSIONS FROM PHOSPHATE
FERTILIZER PLANTS**

**§ 62.4354 Identification of plan—negative
declaration.**

The Kentucky Department for Natural Resources and Environmental Protection certified in a letter dated August 25, 1978, that Kentucky has no

designated facilities in this source category.

Subpart T—Louisiana

SOURCE: 44 FR 54053, Sept. 18, 1979, unless otherwise noted.

**PLAN FOR CONTROL OF DESIGNATED POLLUTANTS FROM EXISTING FACILITIES
(SECTION 111(d) PLAN)**

§ 62.4620 Identification of plan.

(a) Identification of plan: Louisiana Plan for Control of Designated Pollutants from Existing Facilities (111(d) Plan).

(b) The plan was officially submitted as follows:

(1) Control of sulfuric acid mist from sulfuric acid plants, and fluoride emissions from existing facilities at phosphate fertilizer plants, submitted on July 18, 1978, having been adopted by the State November 30, 1977, and letter dated February 16, 1982.

(2) Control of fluoride emissions from existing facilities at primary aluminum plants, submitted on January 12, 1981, having been adopted by the State on December 11, 1980.

(3) Control of total reduced sulfur from existing facilities at kraft pulp mill plants, submitted in December 1979, having been adopted November 27, 1979, and letter dated February 16, 1982.

(c) Designated facilities: The plan applies to existing facilities in the following categories of sources:

- (1) Sulfuric acid plants.
- (2) Phosphate fertilizer plants.
- (3) Primary aluminum plants.
- (4) Kraft pulp mills.

[47 FR 20491–20493, May 12, 1982]

§ 62.4621 Emission standards and compliance schedules.

(a) The requirements of § 60.24(b)(2) of this chapter are not met since the test methods and procedures for determining compliance with the sulfuric acid mist emission standards are not specified.

(b) Emissions from sulfuric acid plants must be measured by the methods in Appendix A to Part 60, or by equivalent or alternative methods as

defined in § 60.2 (t) and (u) respectively.

§ 62.4622 Emission inventories, source surveillance, reports.

(a) The requirements of § 60.25(a) of this chapter are not met since the emission inventories do not provide information as specified in Appendix D to Part 60.

(b) The requirements of § 60.25(c) of this chapter are not met since the plan does not provide for the disclosure of emission data, as correlated with applicable emission standards, to the general public.

(c) *Regulation for public availability of emission data.* (1) Any person who cannot obtain emission data from the agency responsible for making emission data available to the public, as specified in the applicable plan, concerning emissions from any source subject to emission limitations which are part of the approved plan may request that the appropriate Regional Administrator obtain and make public such data. Within 30 days after receipt of any such written request, the Regional Administrator shall require the owner or operator of any such source to submit information within 30 days on the nature and amounts of emissions from such source and any other information as may be deemed necessary by the Regional Administrator to determine whether such source is in compliance with applicable emission limitations or other control measures that are part of the applicable plan.

(2) Commencing after the initial notification by the Regional Administrator pursuant to paragraph (c)(1) of this section, the owner or operator of the source shall maintain records of the nature and amounts of emissions from such source and any other information as may be deemed necessary by the Regional Administrator to determine whether such source is in compliance with applicable emission limitations or other control measures that are part of the plan. The information recorded shall be summarized and reported to the Regional Administrator, on forms furnished by the Regional Administrator, and shall be submitted within 45 days after the end of the reporting period. Reporting peri-

ods are January 1-June 30 and July 1-December 31.

(3) Information recorded by the owner or operator and copies of this summarizing report submitted to the Regional Administrator shall be retained by the owner or operator for 2 years after the date on which the pertinent report is submitted.

(4) Emission data obtained from owners or operators of stationary sources will be correlated with applicable emission limitations and other control measures that are part of the applicable plan and will be available at the appropriate regional office and at other locations in the State designated by the Regional Administrator.

§ 62.4623 Legal authority.

(a) The requirements of § 60.26(a) of this chapter are not met since the plan does not provide adequate legal authority for the State to make emission data, as correlated with applicable emissions standards, available to the general public.

SULFURIC ACID MIST FROM EXISTING SULFURIC ACID PLANTS

§ 62.4624 Identification of sources.

Identification of sources: The plan includes the following sulfuric acid plants:

(1) Agrico Chemical Company in St. James Parish.

(2) Allied Chemical Corporation in Ascension and Iberville Parishes.

(3) Beker Industries in St. Charles Parish.

(4) Cities Services Oil Company in Calcasieu Parish.

(5) E. I. du Pont de Nemours & Company, Inc. in Ascension Parish.

(6) Freeport Chemical Company in St. James Parish.

(7) Freeport Chemical Company in Plaquemines Parish.

(8) Olin Corporation in Caddo Parish.

(9) Stauffer Chemical Company in East Baton Rouge Parish.

[44 FR 54053, Sept. 18, 1979. Redesignated at 47 FR 20491, May 12, 1982]

**FLUORIDE EMISSIONS FROM EXISTING
PHOSPHATE FERTILIZER PLANTS**

§ 62.4625 Identification of sources.

(a) The Plan applies to existing facilities at the following phosphate fertilizer plants:

- (1) Agrico Chemical Company at Donaldsville, Louisiana.
- (2) Allied Chemical Corporation at Geismar, Louisiana.
- (3) Beker Industries at Taft, Louisiana.
- (4) Freeport Chemical at Uncle Sam, Louisiana.
- (5) Monsanto at Luling, Louisiana.

[47 FR 20491, May 12, 1982]

§ 62.4626 Effective date.

(a) The effective date of the portion of the plan applicable to phosphate fertilizer plants is July 12, 1982.

[47 FR 20491, May 12, 1982]

**FLUORIDE EMISSIONS FROM EXISTING
PRIMARY ALUMINUM PLANTS**

§ 62.4627 Identification of sources.

The plan applies to existing facilities at the following primary aluminum plants:

- (1) The Kaiser Plant at Chalmette, Louisiana.
- (2) The CONALCO Plant at Lake Charles, Louisiana.

[47 FR 20492, May 12, 1982]

§ 62.4628 Effective date.

The effective date of this portion of the State's plan is July 12, 1982.

[47 FR 20492, May 12, 1982]

**TOTAL REDUCED SULFUR EMISSIONS
FROM EXISTING KRAFT PULP MILLS**

§ 62.4629 Identification of sources.

The plan applies to existing facilities at the following kraft pulp mill plants:

- (1) Boise at DeRidder, La.
- (2) Boise at Elizabeth, La.
- (3) Continental at Hodge, La.
- (4) Crown-Zellerbach at Bogalusa, La.
- (5) Crown-Zellerbach at St. Francisville, La.
- (6) Georgia-Pacific at Port Hudson, La.

(7) International Paper at Bastrop, La.

(8) Olinkraft at West Monroe, La.

(9) Pineville Kraft at Pineville, La.

(10) Western Kraft at Compton, La.

[47 FR 20493, May 12, 1982]

§ 62.4630 Effective date.

The effective date of the portion of the plan applicable to kraft pulp mills is July 12, 1982.

[47 FR 20493, May 12, 1982]

Subpart U—Maine

**PLAN FOR THE CONTROL OF DESIGNATED
POLLUTANTS FROM EXISTING FACILITIES (SECTION 111(d) PLAN)**

§ 62.4845 Identification of plan.

(a) Identification of plan: Maine Plan for the Control of Designated Pollutants from Existing Plants (Section 111(d) Plan).

(b) The plan was officially submitted as follows:

(1) Control of sulfuric acid mist emissions from existing sulfuric acid production units, submitted on November 10, 1988.

(c) Designated facilities: The plan applies to existing facilities in the following categories of sources:

- (1) Sulfuric acid plants.

[54 FR 22896, May 30, 1989]

**FLUORIDE EMISSIONS FROM EXISTING
PRIMARY ALUMINUM PLANTS**

§ 62.4875 Identification of sources—negative declaration.

The State Department of Environmental Protection submitted on October 3, 1988, a letter certifying that there are no existing primary aluminum reduction plants in the State subject to Part 60, Subpart B of this chapter.

[54 FR 9046 Mar. 3, 1989]

**SULFURIC ACID MIST FROM EXISTING
SULFURIC ACID PLANTS**

§ 62.4900 Identification of sources.

The plan applies to the following existing sulfuric acid plants:

Environmental Protection Agency

§ 62.5351

(a) Delta Chemical in Searsport, Maine.

[54 FR 22896, May 30, 1989]

FLUORIDE EMISSIONS FROM PHOSPHATE FERTILIZER PLANTS

§ 62.4950 Identification of plan—negative declaration.

The State Department of Environmental Protection submitted on April 19, 1978, a letter certifying that there are no existing phosphate fertilizer plants in the state subject to Part 60, Subpart B of this chapter.

[44 FR 54052, Sept 18, 1979. Redesignated at 54 FR 22896, May 30, 1989]

Subpart V—Maryland

AUTHORITY: Clean Air Act, sec. 111(d).

SOURCE: 49 FR 8613, Mar. 8, 1984, unless otherwise noted.

PLAN FOR CONTROL OF DESIGNATED POLLUTANTS FROM EXISTING FACILITIES (SECTION 111(d) PLAN)

§ 62.5100 Identification of plan.

(a) *Identification of plan.* Maryland Plan for Control Designated Pollutants from Existing Facilities (Section 111(d) plan).

(b) The plan was officially submitted as follows:

(1) Control of sulfuric acid mist from sulfuric acid plants, submitted by the Secretary of Health and Mental Hygiene, State of Maryland on August 30, 1978.

(2) Control of TRS emissions from kraft pulp mills, submitted by the Governor of Maryland on May 18, 1981, and approval of a compliance schedule, submitted by the State of Maryland on September 24, 1982.

(3) Control of fluoride emissions from primary aluminum reduction plants, submitted by the Secretary of Health and Mental Hygiene, State of Maryland on January 26, 1984.

(c) *Designated facilities.* The plan applies to existing facilities in the following categories of sources:

- (1) Sulfuric acid plants;
- (2) Kraft pulp mills.
- (3) Primary aluminum reduction plants.

[49 FR 8613, Mar. 8, 1984, as amended at 50 FR 9628, Mar. 11, 1985]

SULFURIC ACID MIST FROM EXISTING SULFURIC ACID PLANTS

§ 62.5101 Identification of sources.

(a) The plan applies to the following existing sulfuric acid plants:

- (1) Olin Corporation, Baltimore City, Maryland.

TOTAL REDUCED SULFUR EMISSIONS FROM EXISTING KRAFT PULP MILLS

§ 62.5102 Identification of sources.

(a) The plan applies to existing facilities at the following kraft pulp mills:

- (1) Westvaco Fine Papers Divisions, Luke, Maryland.

FLUORIDE EMISSIONS FROM PRIMARY ALUMINUM REDUCTION PLANTS

§ 62.5103 Identification of sources.

(a) The plan applies to the following existing primary aluminum reduction plants:

- (1) Eastalco Aluminum Plant, Frederick, Maryland.

[50 FR 9628, Mar. 11, 1985]

Subpart W—Massachusetts

FLUORIDE EMISSIONS FROM PHOSPHATE FERTILIZER PLANTS

§ 62.5350 Identification of plan—negative declaration.

The State Department of Environmental Quality Engineering submitted on April 12, 1978, a letter certifying that there are no existing phosphate fertilizer plants in the state subject to Part 60, Subpart B of this chapter.

[44 FR 54052, Sept 18, 1979]

SULFURIC ACID MIST EMISSIONS FROM EXISTING SULFURIC ACID PLANTS

§ 62.5351 Identification of plan—negative declaration.

On February 18, 1986, the Commonwealth of Massachusetts submitted a letter certifying that there are no existing sulfuric acid plants in the Commonwealth of Massachusetts.

Subpart W—Massachusetts

TOTAL REDUCED SULFUR EMISSIONS FROM EXISTING KRAFT PULP MILLS

§ 62.5375 Identification of plan—negative declaration.

The State Department of Environmental Quality Engineering submitted on July 31, 1979, a letter certifying that there are no existing kraft pulp mills in the State subject to Part 60, Subpart E of this chapter.

[54 FR 9047, Mar. 3, 1989]

FLUORIDE EMISSIONS FROM EXISTING PRIMARY ALUMINUM PLANTS

§ 62.5400 Identification of plan—negative declaration.

The State Department of Environmental Quality Engineering submitted on January 18, 1989, a letter certifying that there are no existing primary aluminum reduction plants in the State subject to Part 60, Subpart B of this chapter.

[54 FR 9047, Mar. 3, 1989]

Subpart X—Michigan

FLUORIDE EMISSIONS FROM PHOSPHATE FERTILIZER PLANTS

§ 62.5600 Identification of plan—negative declaration.

The State Department of Resources submitted on April 18, 1977, a letter certifying that there are no existing phosphate fertilizer plants in the State subject to Part 60, Subpart B of this chapter.

Subpart Y—Minnesota

FLUORIDE EMISSIONS FROM PHOSPHATE FERTILIZER PLANTS

§ 62.5850 Identification of plan—negative declaration.

The State Pollution Control Agency submitted on April 7, 1977, a letter certifying that there are no existing phosphate fertilizer plants in the State subject to Part 60, Subpart B of this chapter.

Subpart Z—Mississippi

PLAN FOR THE CONTROL OF DESIGNATED POLLUTANTS FROM EXISTING FACILITIES (SECTION 111(d) PLAN)

SOURCE: 47 FR 29235, July 6, 1982, unless otherwise noted.

§ 62.6100 Identification of plan.

(a) Identification of plan: Untitled (Section 111(d) Plan).

(b) The plan was officially submitted as follows:

(1) Control of sulfuric acid mist emissions from existing facilities at sulfuric acid plants, submitted on September 17, 1981.

(2) Control of total reduced sulfur emissions from existing kraft pulp mills, submitted on October 30, 1987.

(c) Designated facilities: The plan applies to existing facilities in the following categories of sources:

(1) Sulfuric acid plants.

(2) Phosphate fertilizer plants.

(3) Kraft pulp mills.

[47 FR 29235, July 6, 1982, as amended at 54 FR 7771, Feb. 23, 1989]

SULFURIC ACID MIST FROM EXISTING SULFURIC ACID PLANTS

§ 62.6110 Identification of sources.

The plan applies to existing plants at the following locations: Sulfur burning plant and oleum plant of Mississippi Chemical Corporation in Pascagoula.

FLUORIDE EMISSIONS FROM PHOSPHATE FERTILIZER PLANTS

§ 62.6120 Identification of sources.

The plan applies to existing facilities at the following phosphate fertilizer plants.

(1) Mississippi Chemical Corporation in Pascagoula.

FLUORIDE EMISSIONS FROM PRIMARY ALUMINUM REDUCTION PLANTS

§ 62.6121 Identification of sources—Negative declaration.

The Mississippi Bureau of Pollution Control submitted on March 6, 1985, a letter certifying that there are no existing primary aluminum reduction

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§ 62.6354

plants in the State subject to Part 60, Subpart B of this chapter.

[50 FR 26204, June 25, 1985]

TOTAL REDUCED SULFUR EMISSIONS FROM KRAFT PULP MILLS

§ 62.6122 Identification of sources.

The plan applies to existing facilities at the following kraft pulp mills:

(a) Georgia-Pacific Corporation, Monticello.

(b) International Paper Company, Moss Point.

(c) International Paper Company, Natchez.

(d) International Paper Company, Vicksburg.

[54 FR 7771, Feb. 23, 1989]

Subpart AA—Missouri

SOURCE: 51 FR 8828, Mar. 14, 1986, unless otherwise noted.

PLAN FOR THE CONTROL OF DESIGNATED POLLUTANTS FROM EXISTING FACILI- TIES (SECTION 111(d) PLAN)

§ 62.6350 Identification of plan.

(a) Identification of plan: Missouri Plan for Control of Designated Pollutants from Existing Facilities (Section 111(d) Plan).

(b) The plan was officially submitted as follows:

(1) Control of fluoride emissions from existing facilities at phosphate fertilizer plants, and fluoride emissions from existing facilities at primary aluminum reduction plants, submitted on September 22, 1981, having been adopted by the State on June 17 and June 21, 1981. A letter conveying additional information regarding this plan was submitted on January 3, 1985.

(2) Control of sulfuric acid mist from existing facilities at sulfuric acid production plants, submitted on March 12, 1979, having been adopted by the State in 1967 and 1971. A letter providing additional information regarding this plan was submitted on January 3, 1985.

(c) Designated facilities: The plan applies to existing facilities in the following categories of sources:

(1) Phosphate fertilizer plants.

(2) Primary aluminum reduction plants.

(3) Sulfuric acid production plants.

FLUORIDE EMISSIONS FROM EXISTING PHOSPHATE FERTILIZER PLANTS

§ 62.6351 Identification of sources.

The plan applies to existing facilities at the following phosphate fertilizer plant:

Farmers Chemical Company, Joplin, Missouri

FLOURIDE EMISSIONS FROM EXISTING PRIMARY ALUMINUM REDUCTION PLANTS

§ 62.6352 Identification of sources.

The plan applies to existing facilities at the following primary aluminum reduction plant:

Noranda Aluminum, Inc., New Madrid, Missouri

SULFURIC ACID MIST FROM EXISTING SULFURIC ACID PRODUCTION PLANTS

§ 62.6353 Identification of sources.

The plan applies to existing facilities at the following sulfuric acid production plant:

W.R. Grace and Company, Joplin, Missouri

TOTAL REDUCED SULFUR EMISSIONS FROM EXISTING KRAFT PULP MILLS

§ 62.6354 Identification of plan—negative declaration.

Letter from the Director of the Missouri Department of Natural Resources submitted on May 14, 1982, certifying that there are no kraft pulp mills in the State subject to Part 60, Subpart B of this chapter.

Subpart BB—[Reserved]

Subpart CC—Nebraska

SOURCE: 49 FR 7234, Feb. 28, 1984, unless otherwise noted.

§ 62.6850

**FLUORIDE EMISSIONS FROM EXISTING
PHOSPHATE FERTILIZER PLANTS**

**§ 62.6850 Identification of Plan—Negative
Declaration.**

Letter from the Director of the Department of Environmental Control submitted on May 4, 1977, certifying that there are no phosphate fertilizer plants in the State of Nebraska.

**SULFURIC ACID MIST EMISSIONS FROM
EXISTING SULFURIC ACID PLANTS**

**§ 62.6875 Identification of plan—negative
declaration.**

Letter from the Chief of the Air Pollution Control Division of the Department of Environmental Control submitted on December 9, 1977, certifying that there are no existing sulfuric acid plants in the State of Nebraska.

**TOTAL REDUCED SULFUR EMISSIONS
FROM EXISTING KRAFT PULP MILLS**

**§ 62.6880 Identification of plan—negative
declaration.**

Letter from the Chief of the Air Pollution Control Division of the Department of Environmental Control submitted on March 16, 1984, certifying that there are no existing Kraft pulp mills in the State of Nebraska, subject to Part 60, Subpart B of this chapter.

[49 FR 43058, Oct. 26, 1984]

**FLUORIDE EMISSIONS FROM EXISTING
PRIMARY ALUMINUM REDUCTION PLANTS**

**§ 62.6910 Identification of plan—negative
declaration.**

Letter from the Chief of the Air Pollution Control Division of the Department of Environmental Control submitted on March 16, 1984, certifying that there are no existing primary aluminum reduction plants in the State of Nebraska, subject to Part 60, Subpart B of this chapter.

[49 FR 43058, Oct. 26, 1984]

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Subpart DD—[Reserved]

Subpart EE—New Hampshire

**FLUORIDE EMISSIONS FROM PHOSPHATE
FERTILIZER PLANTS**

**§ 62.7350 Identification of plan—negative
declaration.**

The State Air Pollution Control Agency submitted on November 29, 1978, a letter certifying that there are no existing phosphate fertilizer plants in the state subject to Part 60, Subpart B of this chapter.

[44 FR 54052, Sept 18, 1979]

**SULFURIC ACID MIST EMISSIONS FROM
SULFURIC ACID PRODUCTION UNITS**

**§ 62.7375 Identification of plan—negative
declaration.**

The State Air Pollution Control Agency submitted on November 29, 1978, a letter certifying that there are no existing sulfuric acid plants in the state subject to Part 60, Subpart B of this chapter.

[44 FR 54052, Sept 18, 1979]

**FLUORIDE EMISSIONS FROM EXISTING
PRIMARY ALUMINUM PLANTS**

**§ 62.7400 Identification of sources—negative
declaration.**

The State Air Pollution Control Agency submitted on January 3, 1989, a letter certifying that there are no existing primary aluminum reduction plants in the State subject to Part 60, Subpart B of this chapter.

[54 FR 9047, Mar. 3, 1989]

Subpart FF—New Jersey

**FLUORIDE EMISSIONS FROM PHOSPHATE
FERTILIZER PLANTS**

**§ 62.7600 Identification of plan—negative
declaration**

The New Jersey Department of Environmental Protection submitted, on May 20, 1977, a letter certifying that there are no existing phosphate fertilizer plants in the State subject to Part 60, Subpart B of this chapter.

[44 FR 41180, July 16, 1979]

Environmental Protection Agency

§ 62.8100

TOTAL REDUCED SULFUR EMISSIONS FROM KRAFT PULP MILLS

§ 62.7601 Identification of plan—negative declaration.

The New Jersey Department of Environmental Protection submitted, on October 18, 1979, a letter certifying that there are no existing kraft pulp mills in the State subject to Part 60, Subpart B of this chapter.

[45 FR 80826, Dec. 8, 1980; 46 FR 27342, May 19, 1981]

FLUORIDE EMISSIONS FROM PRIMARY ALUMINUM REDUCTION PLANTS

§ 62.7602 Identification of plan—negative declaration.

The New Jersey Department of Environmental Protection submitted, on September 29, 1980, a letter certifying that there are no existing primary aluminum plants in the State subject to Part 60 Subpart B of this chapter.

[46 FR 30479, June 9, 1981]

Subpart GG—New Mexico

SOURCE: 47 FR 10005, Mar. 9, 1982, unless otherwise noted.

§ 62.7850 Identification of plan.

(a) Title of plan: "State of New Mexico Designated Facility Plan" (§ 111(d)).

(b) The plan was officially submitted as follows:

(1) Sulfuric acid plants on May 15, 1981.

(c) Affected facilities: The plan includes the following facilities:

(1) Sulfuric acid plants.

SULFURIC ACID MIST EMISSIONS FROM SULFURIC ACID PLANTS

§ 62.7851 Identification of sources.

(a) The plan includes the following sources:

(1) Kerr-McGee Nuclear Corporation in McKinley County.

(2) Climax Chemical Corporation in Lea County.

FLUORIDE EMISSIONS FROM PRIMARY ALUMINUM PLANTS

§ 62.7852 Identification of plan—negative declaration.

The New Mexico Environmental Improvement Division and the Albuquerque Air Pollution Control Division submitted letters of July 8, 1980 and September 23, 1980, respectively, certifying that there are no existing primary aluminum plants in the State subject to Part 60, Subpart B of this chapter.

TOTAL REDUCED SULFUR EMISSIONS FROM KRAFT PULP MILLS

§ 62.7853 Identification of plan—negative declaration.

The New Mexico Environmental Improvement Division and the Albuquerque Air Pollution Control Division submitted letters of November 5, 1979 and July 8, 1980, respectively, certifying that there are no existing kraft pulp mills in the State subject to Part 60, Subpart B of this chapter.

FLUORIDE EMISSIONS FROM PHOSPHATE FERTILIZER PLANTS

§ 62.7854 Identification of plan—negative declaration.

The State Department of Health and Social Services submitted on October 31, 1977, a letter certifying that there are no existing phosphate fertilizer plants in the State subject to Part 60, Subpart B of this chapter.

[43 FR 51393, Nov. 3, 1978. Redesignated at 47 FR 10005, Mar. 9, 1982]

Subpart HH—New York

FLUORIDE EMISSIONS FROM PHOSPHATE FERTILIZER PLANTS

§ 62.8100 Identification of plan—negative declaration.

The New York State Department of Environmental Conservation submitted, on May 12, 1977, a letter certifying that there are no existing phosphate fertilizer plants in the State subject to Part 60, Subpart B of this chapter.

[44 FR 41180, July 16, 1979]

SULFURIC ACID MIST EMISSIONS FROM EXISTING SULFURIC ACID PLANTS

§ 62.8102 Identification of plan.

(a) [Reserved]

(b) The plan was officially submitted and approved as follows:

(1) Part 224—"Sulfuric Acid and Nitric Acid Plants" of Title 6 of the New York Code of Rules and Regulations effective May 10, 1984.

(2) Supplemental information submitted on March 29, 1985.

(c) Identification of sources. The plan includes the following plants:

(1) PVS Chemicals, Inc., Buffalo.

(2) Eastman Kodak Company, Rochester.

(d) The plan is approved with the provision that for existing sources any variance or compliance date extension from the provisions of Part 224, "Sulfuric Acid and Nitric Acid Plants," or any text method other than specified in 40 CFR Part 60, Appendix A, approved by the Commissioner of Environmental Conservation must be submitted and approved as a plan revision.

[50 FR 41137, Oct. 9, 1985]

Subpart II—North Carolina

AUTHORITY: Secs. 110(a) and 111(d), Clean Air Act (42 U.S.C. 7410(a) and 7411(d)).

SOURCE: 48 FR 31403, July 8, 1983, unless otherwise noted.

PLAN FOR THE CONTROL OF DESIGNATED POLLUTANTS FROM EXISTING FACILITIES (SECTION 111(d) PLAN)

§ 62.8350 Identification of plan.

(a) *Identification of plan.* North Carolina Designated Facility Plan (Section 111(d) Plan).

(b) *The plan was officially submitted as follows.* (1) Control of sulfuric acid mist emissions from existing sulfuric acid production units, submitted on October 27, 1978, and November 14, 1979.

(2) Control of fluoride emissions from existing primary aluminum plants, submitted on April 16, and August 24, 1981.

(3) Control of total reduced sulfur emissions from existing facilities at

kraft pulp mills, submitted on May 2, 1980, and September 24, 1982.

(4) The following revisions to Title 15 of the North Carolina Administrative Code (15 NCAC) were submitted to EPA on July 18, 1986, following adoption by the North Carolina Environmental Management Commission on November 8, 1984: Revised regulations 2D.0517—Emissions From Plants Producing Sulfuric Acid, 2D.0528—Total Reduced Sulfur From Kraft Pulp Mills, and 2D.0529—Fluoride Emissions From Primary Aluminum Reduction Plants.

(5) A change to regulation 15 NCAC 2D.0528, Total Reduced Sulfur from Kraft Pulp Mills, was submitted to EPA April 14, 1987, following adoption by the North Carolina Environmental Management Commission on April 9, 1987.

(6) Revisions to regulations 15 NCAC 2D.0528(c), (f), (g), and (h)—Total Reduced Sulfur from Kraft Pulp Mills, and 2D.0529 (a) and (c)—Fluoride Emissions from Primary Aluminum Reduction Plants, were submitted by the North Carolina Department of Natural Resources and Community Development on May 2, 1988, following adoption by the North Carolina Environmental Management Commission on April 14, 1988.

(c) *Designated facilities.* The plan applies to existing facilities in the following categories of sources:

(1) Sulfuric acid plants.

(2) Primary aluminum plants.

(3) Kraft pulp mills.

[48 FR 31403, July 8, 1983, as amended at 51 FR 41788, Nov. 19, 1986; 53 FR 31863, Aug. 22, 1988; 53 FR 49882, Dec. 12, 1988]

SULFURIC ACID MIST FROM EXISTING SULFURIC ACID PLANTS

§ 62.8351 Identification of sources.

The plan applies to existing facilities at the following sulfuric acid plants:

(a) Sulfur-burning plants operated by:

(1) Texasgulf Inc. in Beaufort County,

(2) Swift Agricultural Chemical Company in Brunswick County,

(3) USS Agri-Chemicals in Brunswick County,

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§ 62.9100

(4) Wright Chemical Corporation in Columbus County, and

(5) Northeast Chemical Company in New Hanover County.

(b) There are no oleum plants.

(c) There are no bound sulfur feed-stock plants.

FLUORIDE EMISSIONS FROM EXISTING PRIMARY ALUMINUM PLANTS

§ 62.8352 Identification of sources.

(a) The plan applies to the following existing primary aluminum plant facilities.

(1) Two potlines of prebake cells at the Badin (Stanly County) plant of the Aluminum Corporation of America.

TOTAL REDUCED SULFUR EMISSIONS FROM KRAFT PULP MILLS

§ 62.8353 Identification of sources.

The plan applies to existing facilities at the following kraft pulp mills:

(a) Federal Paper Board in Riegelwood,

(b) Hoerner-Waldorf in Roanoke Rapids,

(c) U.S. Plywood-Champion Paper in Canton,

(d) Weyerhaeuser in New Bern, and

(e) Weyerhaeuser in Plymouth.

Subpart JJ—[Reserved]

Subpart KK—Ohio

FLUORIDE EMISSIONS FROM PHOSPHATE FERTILIZER PLANTS

§ 62.8850 Identification of plan—negative declaration.

The Ohio Environmental Protection Agency submitted on December 1, 1977, (resubmitted on April 1, 1985, and April 25, 1985) a letter certifying that there are no existing phosphate fertilizer plants in the State subject to Part 60, Subpart B of this chapter.

[50 FR 41137, Oct. 9, 1985]

TOTAL REDUCED SULFUR EMISSIONS FROM KRAFT PULP MILLS

§ 62.8860 Identification of Plan—Disapproval.

On December 7, 1984, and April 23, 1986, Ohio submitted its plan for controlling total reduced sulfur from existing kraft pulp mills, including Rules 3745-73-01, 02, 03, and 04. The plan is being disapproved because:

(a) The requirements of § 60.24(f) of this chapter are not met because the State failed to justify the application of emission standards less stringent than the Federal emission standards. Additionally, USEPA does not have a bubble policy applicable to 111(d) plans.

(b) The plan does not contain monitoring requirements to ensure proper operation and maintenance of the affected facility as required by § 60.25(b) of this chapter.

[55 FR 19884, May 14, 1990]

Subpart LL—Oklahoma

PLAN FOR THE CONTROL OF DESIGNATED POLLUTANTS FROM EXISTING FACILITIES (SECTION 111(d) PLAN)

§ 62.9100 Identification of plan.

(a) *Identification of plan:* Oklahoma Plan for Control of Designated Pollutants from Existing Facilities (Section 111(d) Plan).

(b) The plan was officially submitted as follows:

(1) Control of sulfuric acid mist from existing sulfuric acid production plants submitted on December 5, 1985, with the corresponding regulation submitted by the Governor of Oklahoma on March 31, 1986.

(2) Control of total reduced sulfur from existing kraft pulp mills was submitted on November 17, 1987, and supplemented on June 1, 1988.

(c) Designated facilities: The plan applies to existing facilities in the following categories of sources.

(1) Sulfuric acid production plants.

(2) Kraft pulp mills.

[52 FR 3229, Feb. 3, 1987, as amended at 54 FR 24905, June 12, 1989]

§ 62.9110

SULFURIC ACID MIST FROM EXISTING SULFURIC ACID PLANTS

§ 62.9110 Identification of sources.

(a) *Identification of sources:* The plan includes the following sulfuric acid production plants.

(1) National Zinc Co. in Bartlesville, Oklahoma.

(2) Tulsa Chemical Co. in Tulsa, Oklahoma.

[52 FR 3230, Feb. 3, 1987]

FLUORIDE EMISSIONS FROM PHOSPHATE FERTILIZER PLANTS

§ 62.9120 Identification of plan—negative declaration.

The State Department of Health submitted on October 25, 1977, a letter certifying that there are no existing phosphate fertilizer plants in the State subject to Part 60, Subpart B of this chapter.

[43 FR 51393, Nov. 3, 1978. Redesignated at 52 FR 3229, Feb. 3, 1987]

FLUORIDE EMISSIONS FROM PRIMARY ALUMINUM PLANTS

§ 62.9130 Identification of plan—negative declaration.

The Oklahoma State Department of Health submitted a letter on March 3, 1983, certifying that there are no existing primary aluminum reduction plants in the State of Oklahoma subject to 40 CFR Part 60, Subpart B, of this chapter.

[48 FR 29854, June 29, 1983. Redesignated at 52 FR 3229, Feb. 3, 1987]

TOTAL REDUCED SULFUR FROM EXISTING KRAFT PULP MILLS

§ 62.9140 Identification of source.

The plan includes the following kraft pulp mill:

(a) Weyerhaeuser Paper Company in Valliant, Oklahoma.

[54 FR 24905, June 12, 1989]

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Subpart MM—Oregon

PLAN FOR THE CONTROL OF DESIGNATED POLLUTANTS FROM EXISTING FACIL- ITIES (SECTION 111(d) PLAN)

§ 62.9350 Identification of plan.

(a) *Identification of plan:* Oregon Designated Facility Plan (Section 111(d) Plan).

(b) The plan was officially submitted as follows:

(1) Control of fluoride emissions from phosphate fertilizer plants, submitted by the Oregon State Department of Environmental Quality on June 1, 1977.

(2) Control of sulfuric acid mist emissions from sulfuric acid production units, submitted by the Oregon State Department of Environmental Quality on January 27, 1978.

(3) Control of fluoride emissions from primary aluminum reduction plants, submitted by the Oregon State Department of Environmental Quality on January 13, 1981 and August 9, 1982.

(c) *Designated facilities:* The plan applies to existing facilities in the following categories of sources:

(1) Phosphate fertilizer plants.

(2) Sulfuric acid production units.

(3) Primary aluminum reduction plants.

[48 FR 11118, Mar. 16, 1983]

FLUORIDE EMISSIONS FROM PRIMARY ALUMINUM REDUCTION PLANTS

§ 62.9360 Identification of sources.

The plan applies to existing facilities at the following primary aluminum reduction plants:

(a) Reynolds Metals Company in Troutdale, Oregon

(b) Martin-Marietta in The Dalles, Oregon.

[48 FR 11118, Mar. 16, 1983]

FLUORIDE EMISSIONS FROM PHOSPHATE FERTILIZER PLANTS

§ 62.9500 Identification of sources.

The Oregon State Department of Environmental Quality submitted on June 1, 1977, certification that there are no existing phosphate fertilizer

plants in the State subject to Part 60, Subpart B of this chapter.

[44 FR 76281, Dec. 26, 1979. Redesignated and amended at 48 FR 11118, Mar. 16, 1983]

SULFURIC ACID MIST EMISSIONS FROM SULFURIC ACID PRODUCTION UNITS

§ 62.9501 Identification of sources.

The Oregon State Department of Environmental Quality submitted on January 27, 1978, certification that there are no existing sulfuric acid plants in the State subject to Part 60, Subpart B of this chapter.

[44 FR 76281, Dec. 26, 1979. Redesignated and amended at 48 FR 11118, Mar. 16, 1983]

Subpart NN—Pennsylvania

FLUORIDE EMISSIONS FROM PHOSPHATE FERTILIZER PLANTS

§ 62.9600 Identification of plan—negative declaration.

(a) The Pennsylvania Department of Environmental Resources submitted on December 1, 1977, a letter certifying that there are no existing phosphate fertilizer plants in the State subject to Part 60, Subpart B of this chapter.

(b) The Allegheny County Bureau of Air Pollution Control submitted a letter on August 18, 1978 certifying that there are no phosphate fertilizer plants in the County subject to part 60, Subpart B of this chapter.

(c) The City of Philadelphia Air Management Services submitted on February 22, 1985, a letter certifying that there are no existing phosphate fertilizer plants in the City subject to Part 60, Subpart B of this chapter.

[47 FR 5900, Feb. 9, 1982, and 50 FR 47734, Nov. 20, 1985]

SULFURIC ACID MIST EMISSIONS FROM EXISTING SULFURIC ACID PLANTS

§ 62.9601 Identification of plan.

(a) The Allegheny County Bureau of Air Pollution Control submitted a letter on August 18, 1978 certifying that there are no sulfuric acid plants in the County subject to Part 60, Subpart B of this chapter.

(b) A plan for the control of sulfuric acid mist emissions from existing sulfuric acid plants in the Commonwealth of Pennsylvania, submitted on May 30, 1978 and supplemented on August 17, 1981.

(c) The City of Philadelphia Air Management Services submitted on February 22, 1985, a letter certifying that there are no existing sulfuric acid plants in the City subject to Part 60, Subpart B of this chapter.

[47 FR 5900, Feb. 9, 1982, and 50 FR 47735, Nov. 20, 1985]

TOTAL REDUCED SULFUR EMISSIONS FROM EXISTING KRAFT PULP MILLS

§ 62.9610 Identification of plan—negative declaration

(a) The Allegheny County Bureau of Air Pollution Control submitted a letter on February 14, 1985, certifying that there are no kraft pulp mills in the County subject to Part 60, Subpart B of this chapter.

(b) The City of Philadelphia Air Management Services submitted on February 22, 1985, a letter certifying that there are no existing kraft pulp mills in the City subject to Part 60, Subpart B of this chapter.

[50 FR 47735, Nov. 20, 1985]

FLUORIDE EMISSIONS FROM EXISTING PRIMARY ALUMINUM PLANTS

§ 62.9620 Identification of plan—negative declaration.

The Secretary, Department of Environmental Resources submitted on November 3, 1980, a letter certifying that there are no primary aluminum plants in the Commonwealth of Pennsylvania subject to Part 60, Subpart B of this chapter.

[46 FR 41783, Aug. 18, 1981]

Subpart OO—Rhode Island

FLUORIDE EMISSIONS FROM PHOSPHATE FERTILIZER PLANTS

§ 62.9850 Identification of plan—negative declaration.

The State Department of Environmental Management submitted on No-

venber 14, 1977, a letter certifying that there are no existing phosphate fertilizer plants in the state subject to Part 60, Subpart B of this chapter.

[44 FR 54052, Sept. 18, 1979]

**SULFURIC ACID MIST EMISSIONS FROM
SULFURIC ACID PRODUCTION UNITS**

**§ 62.9875 Identification of plan—negative
declaration.**

The State Department of Environmental Management submitted on November 14, 1977, a letter certifying that there are no existing sulfuric acid plants in the state subject to Part 60, Subpart B of this chapter.

[44 FR 54052, Sept. 18, 1979]

**TOTAL REDUCED SULFUR EMISSIONS
FROM EXISTING KRAFT PULP MILLS**

**§ 62.9900 Identification of plan—negative
declaration.**

The State Department of Environmental Management submitted on July 26, 1979, a letter certifying that there are no existing kraft pulp mills in the State subject to Part 60, Subpart B of this chapter.

[54 FR 9047, Mar. 3, 1989]

**FLUORIDE EMISSIONS FROM EXISTING
PRIMARY ALUMINUM PLANTS**

**§ 62.9950 Identification of plan—negative
declaration.**

The State Department of Environmental Management submitted on December 8, 1989, a letter certifying that there are no existing primary aluminum reduction plants in the State subject to Part 60, Subpart B of this chapter.

[54 FR 9047, Mar. 3, 1989]

Subpart PP—South Carolina

**PLAN FOR THE CONTROL OF DESIGNATED
POLLUTANTS FROM EXISTING FACILITIES
(SECTION 111(d) PLAN)**

SOURCE: 47 FR 29236, July 6, 1982, unless otherwise noted.

§ 62.10100 Identification of plan.

(a) Identification of Plan: South Carolina Designated Facility Plan (Section 111(d) Plan).

(b) The plan was officially submitted as follows: (1) Implementation Plan for Control of Designated Pollutants, including sulfuric acid mist from sulfuric acid plants and total reduced sulfur from kraft pulp mills, submitted on December 22, 1981.

(2) A revision to South Carolina's 111(d) plan for total reduced sulfur which was submitted on December 12, 1984. This revision approved an alternate emission limit for the digesters and an extended compliance schedule for the evaporators at Stone Container Corporation.

(c) Designated facilities: The plan applies to existing facilities in the following categories sources:

- (1) Sulfuric acid plants.
- (2) Kraft Pulp Mills.

[47 FR 29236, July 6, 1982, as amended at 50 FR 33037, Aug. 16, 1985]

**SULFURIC ACID MIST FROM SULFURIC
ACID PLANTS**

§ 62.10110 Identification of sources.

The plan applies to existing plants at the following locations:

- (1) Sulfur-burning plants of W. R. Grace and Company's plant in Charleston.
- (2) There are no bound sulfur or oleum plants.

**TOTAL REDUCED SULFUR EMISSIONS
FROM KRAFT PULP MILLS**

§ 62.10120 Identification of sources.

The plan applies to existing facilities at the following Kraft Pulp Mills:

- (1) Westvaco in North Charleston.
- (2) International Paper Company in Georgetown.
- (3) Bowater Carolina Company in Catawba.
- (4) Stone Container Corporation in Florence.

[47 FR 29236, July 6, 1982, as amended at 50 FR 33037, Aug. 16, 1985]

Environmental Protection Agency

§ 62.11425

FLUORIDE EMISSIONS FROM PHOSPHATE FERTILIZER PLANTS

[47 FR 25336, June 11, 1982; 48 FR 10316, Mar. 11, 1983]

§ 62.10130 Identification of plan—negative declaration.

The South Carolina Department of Health and Environmental Control submitted on November 2, 1977, a letter certifying that there are no existing phosphate fertilizer plants in the State subject to Part 60, Subpart B, of this chapter.

FLUORIDE EMISSIONS FROM EXISTING PRIMARY ALUMINUM REDUCTION PLANTS

§ 62.10140 Identification of plan—negative declaration.

The South Carolina Department of Health and Environmental Control submitted on May 3, 1983, a letter certifying that there are no existing primary aluminum plants in the State which are subject to Part 60 Subpart B of this chapter.

[50 FR 33037, Aug. 16, 1985]

Subpart RR—Tennessee

FLUORIDE EMISSIONS FROM PHOSPHATE FERTILIZER PLANTS

§ 62.10602 Identification of sources—Negative declaration.

The Tennessee Department of Health and Environment on April 4, 1985, submitted a letter certifying that there are no existing phosphate fertilizer plants in the State subject to Part 60, Subpart B of this chapter.

[50 FR 26204, June 25, 1985]

Subpart SS—[Reserved]

Subpart TT—Utah

FLUORIDES FROM EXISTING PHOSPHATE FERTILIZER PLANTS

§ 62.11100 Identification of plan.

(a) *Title of plan:* "Control of Fluorides from Existing Phosphate Plants".

(b) The plan was officially submitted on March 1, 1982.

(c) *Identification of Source:* The plan includes the Chevron Chemical Company's phosphate fertilizer plant in Magna, Utah.

Subpart UU—Vermont

FLUORIDE EMISSIONS FROM PHOSPHATE FERTILIZER PLANTS

§ 62.11350 Identification of plan—negative declaration.

The State Agency of Environmental Conservation submitted on April 11, 1978, a letter certifying that there are no existing phosphate fertilizer plants in the state subject to Part 60, Subpart B of this chapter.

[44 FR 54052, Sept. 18, 1979]

SULFURIC ACID MIST EMISSIONS FROM SULFURIC ACID PRODUCTION UNITS

§ 62.11375 Identification of plan—negative declaration.

The State Agency of Environmental Conservation submitted on April 11, 1978, a letter certifying that there are no existing sulfuric acid plants in the state subject to Part 60, Subpart B of this chapter.

[44 FR 54053, Sept. 18, 1979]

TOTAL REDUCED SULFUR EMISSIONS FROM EXISTING KRAFT PULP MILLS

§ 62.11400 Identification of plan—negative declaration.

The State Agency of Environmental Conservation submitted on August 4, 1979, a letter certifying that there are no existing kraft pulp mills in the State subject to Part 60, Subject B of this chapter.

[54 FR 9047, Mar. 3, 1989]

FLUORIDE EMISSIONS FROM EXISTING PRIMARY ALUMINUM PLANTS

§ 62.11425 Identification of plan—negative declaration.

The State Agency of Environmental Conservation submitted on January 4, 1989, a letter certifying that there are no existing primary aluminum reduction plants in the State subject to Part 60, Subpart B of this chapter.

[54 FR 9047, Mar. 3, 1989]

Subpart VV—Virginia

FLUORIDE EMISSIONS FROM PHOSPHATE FERTILIZER PLANTS

§ 62.11600 Identification of plan—negative declaration.

The Secretary of Commerce and Resources, Office of the Governor submitted on May 13, 1977, a letter certifying that there are no existing phosphate fertilizer plants in the State subject to Part 60, Subpart B of this chapter.

[45 FR 43412, June 27, 1980]

SULFURIC ACID MIST EMISSIONS FROM EXISTING SULFURIC ACID PLANTS

§ 62.11601 Identification of plan.

(a) Title of plan: Commonwealth of Virginia State Implementation Plan under section 111(d) of the Clean Air Act for the Designated Facility—Sulfuric Acid Plants.

(b) The plan was officially submitted by the Secretary of Commerce and Resources, Commonwealth of Virginia, on September 29, 1978.

(c) The plan is approved except that no action is being taken on the monitoring and source surveillance procedures (section 4.04 of the Virginia Air Quality Control Regulations) and the notification, records and reporting procedures (section 4.05 of the Virginia Air Quality Control Regulations) which will be acted on separately under Part 52.

(d) Identification of sources: The plan includes the following sulfuric acid plants:

Allied Chemical, Hopewell
Allied Chemical, Front Royal
Du Pont, James River
Smith Douglas, Chesapeake
U.S. Army Ammo Plant, Radford
Weaver Fertilizer, Norfolk

(e) A variance issued to the E. I. du Pont de Nemours and Company James River Sulfuric Acid Plant located in Chesterfield County, Virginia exempting the plant from section 4.51(c)(2) until December 15, 1981, submitted on October 21, 1980 by the Virginia Secretary of Commerce and Resources.

[46 FR 55973, Nov. 13, 1981, as amended at 46 FR 55975, Nov. 13, 1981]

§ 62.11602 Emission standards and compliance schedules.

(a) The requirements of § 60.25 (b) and (c) of this chapter are not met since EPA is taking separate action on §§ 4.04 and 4.05 and Appendix J under Part 52 of this title.

[46 FR 55974, Nov. 13, 1981]

TOTAL REDUCED SULFUR EMISSIONS FROM EXISTING KRAFT PULP MILLS

§§ 62.11610—62.11619 [Reserved—plan not submitted]

FLUORIDE EMISSIONS FROM EXISTING PRIMARY ALUMINUM PLANTS

§ 62.11620 Identification of plan—negative declaration.

The Commonwealth of Virginia, Office of the Governor, submitted on July 9, 1980, a letter certifying that there are no designated facilities in the Commonwealth subject to the emission guidelines set forth in the Final Guideline Document for the Control of Fluoride Emissions from Existing Primary Aluminum Plants.

[46 FR 41783, Aug. 18, 1981]

Subpart WW—Washington

FLUORIDE EMISSIONS FROM PHOSPHATE FERTILIZER PLANTS

§ 62.11850 Identification of plan—negative declaration.

The Washington State Department of Ecology submitted on August 29, 1979, certification that there are no existing phosphate fertilizer plants in the State subject to Part 60, Subpart B of this chapter.

[44 FR 76281, Dec. 26, 1979]

Subpart XX—West Virginia

FLUORIDE EMISSIONS FROM PHOSPHATE FERTILIZER PLANTS

§ 62.12100 Identification of plan—negative declaration.

The West Virginia Air Pollution Control Commission submitted on October 25, 1977, a letter certifying that there are no existing phosphate fertil-

izer plants in the State subject to Part 60, Subpart B of this chapter.

[45 FR 43412, June 27, 1980]

Subpart YY—Wisconsin

FLUORIDE EMISSIONS FROM PHOSPHATE FERTILIZER PLANTS

§ 62.12350 Identification of plan—negative declaration.

The State Department of Natural Resources submitted on May 24, 1977, a letter certifying that there are no existing phosphate fertilizer plants in the State subject to Part 60, Subpart B of this chapter.

Subparts ZZ—AAA—[Reserved]

Subpart BBB—Puerto Rico

FLUORIDE EMISSIONS FROM PHOSPHATE FERTILIZER PLANTS

§ 62.13100 Identification of plan—negative declaration

The Commonwealth Environmental Quality Board submitted, on January 31, 1978, a letter certifying that there are no existing phosphate fertilizer plants in Commonwealth subject to Part 60, Subpart B of this chapter.

[44 FR 41180, July 16, 1979]

SULFURIC ACID MIST EMISSIONS FROM SULFURIC ACID PLANTS

§ 62.13101 Identification of plan—negative declaration.

The Commonwealth Environmental Quality Board submitted, on January 31, 1978, a letter certifying that there are no existing sulfuric acid plants in the Commonwealth subject to Part 60, Subpart B of this chapter.

[45 FR 37432, June 3, 1980; 46 FR 27342, May 19, 1981]

FLUORIDE EMISSIONS FROM PRIMARY ALUMINUM REDUCTION PLANTS

§ 62.13102 Identification of plan—negative declaration.

The Commonwealth of Puerto Rico submitted on April 28, 1981, a letter certifying that there are no existing primary aluminum plants in the Com-

monwealth subject to Part 60 Subpart B of this chapter.

[46 FR 43834, Sept. 1, 1981]

TOTAL REDUCED SULFUR FROM KRAFT PULP MILLS

§ 62.13103 Identification of plan—negative declaration.

The Commonwealth of Puerto Rico submitted on April 28, 1981, a letter certifying that there are no existing kraft pulp mills in the Commonwealth subject to Part 60 Subpart B of this chapter.

[46 FR 43834, Sept. 1, 1981]

Subpart CCC—Virgin Islands

FLUORIDE EMISSIONS FROM PHOSPHATE FERTILIZER PLANTS

§ 62.13350 Identification of plan—negative declaration

The Territory Department of Conservation and Cultural Affairs submitted, on November 3, 1977, a letter certifying that there are no existing phosphate fertilizer plants in the Territory subject to Part 60, Subpart B of this chapter.

[44 FR 41181, July 16, 1979]

SULFURIC ACID MIST EMISSIONS FROM SULFURIC ACID PLANTS

§ 62.13351 Identification of plan—negative declaration.

The Territory Department of Conservation and Cultural Affairs submitted, on November 8, 1977, a letter certifying that there are no existing sulfuric acid plants in the Territory subject to Part 60, Subpart B of this chapter.

[45 FR 37432, June 3, 1980; 46 FR 27342, May 19, 1981]

TOTAL REDUCED SULFUR EMISSIONS FROM KRAFT PULP MILLS

§ 62.13352 Identification of plan—negative declaration.

The Virgin Islands Department of Conservation and Cultural Affairs submitted, on July 31, 1979, a letter certifying that there are no existing kraft

§ 62.13353

pulp mills in the Territory subject to Part 60, Subpart B of this chapter.

[45 FR 80826, Dec. 8, 1980; 46 FR 27342, May 19, 1981]

FLUORIDE EMISSIONS FROM PRIMARY ALUMINUM REDUCTION PLANTS

§ 62.13353 Identification of plan—negative declaration.

The Virgin Islands Department of Conservation and Cultural Affairs submitted, on July 21, 1980, a letter certifying that there are no primary aluminum plants in the Territory subject to Part 60, Subpart B of this chapter.

[46 FR 30497, June 9, 1981]

PART 65—DELAYED COMPLIANCE ORDERS

Subpart A—General Provisions

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Subparts B—C—[Reserved]

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- 65.70 Federal delayed compliance orders issued under section 113(d) (1), (3), (4) and (5) of the Act.
- 65.71—65.72 [Reserved]

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- 65.140 [Reserved]
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- 65.150 Federal delayed compliance orders issued under section 113(d) (1), (3), (4) and (5) of the Act.
- 65.151—65.152 [Reserved]

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- 65.180 Federal delayed compliance orders issued under section 113(d) (1), (3), (4) and (5) of the Act.
- 65.181—65.182 [Reserved]

Subparts P—S—[Reserved]

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- 65.230 [Reserved]
- 65.231 EPA approval of State delayed compliance orders issued to major stationary sources.
- 65.232 [Reserved]

Subpart U—[Reserved]

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- 65.250 Federal delayed compliance orders issued under section 113(d) (1), (3), (4) and (5) of the Act.
- 65.251—65.252 [Reserved]

Subpart W—Massachusetts

- 65.260 Federal delayed compliance orders issued under section 113(d) (1), (3), (4) and (5) of the Act.
- 65.261—65.262 [Reserved]

Subpart X—Michigan

- 65.270 Federal delayed compliance orders issued under section 113(d) (1), (3), (4) and (5) of the Act.
- 65.271 EPA approval of State delayed compliance orders issued to major stationary sources.
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Subparts Y—Z—[Reserved]

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- 65.300 [Reserved]
- 65.301 EPA approval of State delayed compliance orders issued to major stationary sources.
- 65.302 [Reserved]

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Subparts BB-EE—[Reserved]

Subpart FF—New Jersey

- 65.350 Federal delayed compliance orders issued under section 113(d) (1), (3), (4) and (5) of the Act.
65.351—65.352 [Reserved]

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Subpart NN—Pennsylvania

- 65.430 [Reserved]
65.431 EPA approval of State delayed compliance orders issued to major stationary sources.
65.432 [Reserved]

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- 65.470 [Reserved]
65.471 EPA approval of State delayed compliance orders issued to major stationary sources.
65.472 [Reserved]

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- 65.480 [Reserved]
65.481 EPA approval of State delayed compliance orders issued to major stationary sources.
65.482 [Reserved]

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- 65.510 Federal delayed compliance orders issued under section 113(d) (1), (3), (4) and (5) of the Act.
65.511—65.512 [Reserved]

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- 65.520 [Reserved]
65.521 EPA approval of State delayed compliance orders issued to major stationary sources.
65.522 [Reserved]

Subpart XX—[Reserved]

Subpart YY—Wisconsin

- 65.540 Federal delayed compliance orders issued under section 113(d) (1), (3), (4) and (5) of the Act.
65.541—65.542 [Reserved]

Sec.

Subpart ZZ—[Reserved]

Subpart AAA—Guam

- 65.560 Federal delayed compliance orders issued under section 113(d) (1), (3), (4) and (5) of the Act.
65.561—65.562 [Reserved]

Subparts BBB-EEE—[Reserved]

AUTHORITY: Secs. 113 and 301 of the Clean Air Act, as amended, 42 U.S.C. 7413 and 7601.

SOURCE: 43 FR 44523, Sept. 28, 1978, unless otherwise noted.

Subpart A—General Provisions

§ 65.01 Definitions.

All terms used in this part but not defined herein shall have the meaning given them in the Clean Air Act and in Part 51 of this chapter.

(a) "Act" shall mean the Clean Air Act, as amended, 42 U.S.C. 7401 et seq.

(b) "Administrator" shall mean the Administrator of the U.S. Environmental Protection Agency, or his authorized delegatee.

(c) "Regional Administrator" shall mean the Regional Administrator for an EPA regional office whose duties include those set forth at 40 CFR 1.41, or his authorized delegatee.

(d) "Major stationary source" shall mean any stationary source which directly emits, or has the potential to emit, 100 tons per year or more of any air pollutant for which a national ambient air quality standard under section 109 of the Act is in effect (including any major stationary source of fugitive emissions of any such pollutant, as determined by rule by the Administrator).

(e) "Delayed compliance order" shall mean an order issued by a State or by the Administrator to a stationary source which postpones the date by which the source is required to comply with any requirement contained in the applicable State implementation plan.

(f) The term "Federal delayed compliance order" shall mean a delayed compliance order issued by the Administrator under section 113(d) (1), (3), (4) or (5) of the Act.

(g) The term "State delayed compliance order" shall mean a delayed compliance order issued by a State or by a political subdivision of a State.

(h) "State implementation plan" shall mean the plan, including the most recent revision thereof, which has been approved or promulgated by the Administrator under section 110 of the Act, and which implements the requirements of section 110.

(i) "Stationary source" shall mean any stationary building, facility, equipment, installation, or operation (or combination thereof) which is located on one or more contiguous or adjacent properties and which is owned or operated by the same person (or by persons under common control), and which emits an air pollutant for which a national ambient air quality standard promulgated under section 109 of the Act is in effect.

[43 FR 44523, Sept. 28, 1978, as amended at 48 FR 44744, Sept. 29, 1983]

§ 65.02 Introduction.

(a) Section 113(d) of the Act authorizes the Administrator to issue to certain sources delayed compliance orders permitting a delay in compliance with applicable regulations contained in a State implementation plan. All orders issued by the Administrator under section 113(d) (1), (3), (4) or (5) of the Act will be issued in accordance with the procedures set forth in this part. All such federal delayed compliance orders will be summarized under the subpart corresponding to the State in which the affected source is located.

(b) Section 113(d)(2) of the Act provides that State orders issued to major stationary sources which permit a delay in compliance with applicable State implementation plan regulations must be approved by the Administrator before they have the consequences given delayed compliance orders by section 113(d) of the Act. The Administrator will approve or disapprove, in accordance with the procedures of this part, State delayed compliance orders for major stationary sources which have been submitted to EPA for review under section 113(d)(2) of the Act and § 65.07. All State delayed compliance orders approved or disapproved by the Administrator will be

summarized under the subpart corresponding to the State in which the affected source is located.

(c) Section 113(d)(2) of the Act provides that State orders issued to sources other than major stationary sources which permit a delay in compliance with applicable State implementation plan regulations shall cease to be effective as delayed compliance orders under section 113(d) of the Act upon disapproval by the Administrator. The Administrator will promulgate any such disapproval in accordance with the procedures set forth in this part. All such disapprovals will be summarized in the subpart corresponding to the State in which the affected source is located.

(d) The Administrator will issue or approve a delayed compliance order only if it meets all applicable requirements of section 113(d) of the Act.

[43 FR 44523, Sept. 28, 1978, as amended at 48 FR 44745, Sept. 29, 1983]

§ 65.03 Effect of delayed compliance orders

(a) *EPA approved State delayed compliance orders to major stationary sources.* A State delayed compliance order issued to a major stationary source, upon approval by the Administrator under section 113(d)(2) of the Act in accordance with this part, shall have the following effects:

(1) The order shall become part of the applicable State implementation plan for the purposes of sections 110, 304, and 307 of the Act; and

(2) The order shall preclude enforcement action against the source under sections 113 and 304 of the Act for violations of the State implementation plan provision(s) covered by the order during the period the order is in effect and the source is complying with the terms of the order.

(b) *State delayed compliance orders to nonmajor stationary sources.* A State delayed compliance order issued to a nonmajor stationary source shall, upon issuance by the State, have the effects specified in paragraph (a) of this section, and will be considered an approved order for the purposes of the part, unless the Administrator disap-

proves the order under section 113(d)(2) of the Act.

(c) *State delayed compliance orders not approved by EPA.* A State delayed compliance order which has not been approved by the Administrator under this part does not have the effects specified by paragraph (a) of this section and § 65.04(e).

(d) *Federal delayed compliance order.* A Federal delayed compliance order issued by the Administrator under section 113(d) (1), (3), (4) or (5) of the Act in accordance with this part precludes enforcement action against the source by EPA under section 113 and by citizens under section 304 of the Act for violations of the State implementation plan provision(s) covered by the order during the period the order is in effect and the source is complying with the terms of the order.

(e) *Source responsibilities and liabilities.* (1) The obligations of a source subject to a delayed compliance order include, but are not limited to:

(i) Compliance with the provisions of the delayed compliance order;

(ii) Compliance with applicable State, local, and Federal requirements not covered by the order;

(iii) Compliance with any requirement imposed pursuant to section 303 of the Act, or pursuant to an emergency plan provision contained in an applicable State implementation plan, during conditions which might create a period of imminent and substantial endangerment to the health of persons.

(2) Compliance with a delayed compliance order promulgated or approved under this part does not preclude enforcement actions initiated under applicable State law for violations of the State implementation plan provisions covered by the order.

[43 FR 44523, Sept. 28, 1978, as amended at 48 FR 44745, Sept. 29, 1983]

§ 65.04 Public participation.

(a) *Federal delayed compliance order.* (1) The appropriate EPA Regional Administrator will provide the public an opportunity to submit written comments and to request a public hearing on any proposed Federal delayed compliance order prior to its issuance. This opportunity and the con-

tents of the proposed Federal order will be published as a notice of proposed rulemaking in the FEDERAL REGISTER. The notice of proposed rulemaking will also provide 30 days notice to the State as required by section 113(d)(1) of the Act, unless notice to the State was given earlier. The public comment period will extend no less than 30 days from the date of the notice.

(2) A period of at least 15 days from publication of the notice described in paragraph (a)(1) of this section will be allowed for receipt of requests for a public hearing. A public hearing will be held if the EPA Regional Administrator determines that there is significant public interest in the hearing. If a hearing is held, it shall take place no less than 21 days following publication of notice in the FEDERAL REGISTER containing, at a minimum, the date, time, place, and subject of the hearing.

(3) If the EPA Regional Administrator anticipates substantial public interest in a hearing on a proposed Federal delayed compliance order, a public hearing will be scheduled by the notice described in paragraph (a)(1) of this section, and will take place no less than 21 days following publication of this notice.

(4) The record of a public hearing will remain open for 10 days after the close of the hearing (or for such other period as set forth in the notice of the hearing). Hearing participants may submit additional information to the record during this time.

(5) Due consideration will be given by EPA to all relevant public comment, whether submitted as written comment or adduced at a hearing, and where appropriate the proposed delayed compliance order will be modified or withdrawn in view of such comments.

(6) If, after the 30 day public comment period and the close of the record of any public hearing on the proposed delayed compliance order, the Administrator determines that the proposed order should be issued, he will promulgate the order in this part, through a FEDERAL REGISTER notice of final rulemaking. If the Administrator determines that a proposed Federal delayed compliance order should not

be issued, he will publish a notice in the **FEDERAL REGISTER** withdrawing the proposal of the order.

(b) *EPA approval or disapproval of a State delayed compliance order.* (1) The Regional Administrator will provide the public with an opportunity to submit written comments on whether EPA should approve a State delayed compliance order for a major stationary source prior to EPA's final approval or disapproval of the order. Notice of the opportunity to comment will be published in the **FEDERAL REGISTER**. The public comment period will extend for a minimum of 30 days.

(2) The notice described in paragraph (b)(1) of this section will contain, at a minimum, a summary of the contents of the State order under consideration, including the name and location of the source, the State implementation plan regulation(s) covered by the order, a brief summary of the schedule and plan for compliance required by the order, and the date specified for final compliance with the regulation.

(3) Due consideration will be given to all relevant written public comments and, where appropriate, any proposed EPA action on the order will be modified in view of such comments.

(4) Following the public comment period, the Administrator will promulgate in this part through a **FEDERAL REGISTER** notice of final rulemaking EPA's final approval or disapproval of the State order under consideration.

(5) The procedures specified in this subsection shall apply to the Administrator's disapproval of a delayed compliance order issued by a State to a source other than a major stationary source.

(c) *Revisions of delayed compliance orders.* (1) EPA will follow the procedures specified in paragraph (a) of this section prior to any revision of a Federal delayed compliance order.

(2) EPA will follow the procedures specified in paragraph (b) of this section prior to approval of disapproval of any revision submitted by a State or local agency to an EPA approved State delayed compliance order.

(d) *Revocation of delayed compliance orders.* EPA will follow the procedures specified in paragraph (a) of this

section prior to the revocation of a delayed compliance order under section 113(d)(9)(B) of the Act.

(e) *Judicial review.* Publication of the notice of final rulemaking specified in paragraph (a)(6) and (b)(4) of this section will constitute final Agency action for the purposes of judicial review under section 307(b) of the Act.

§ 65.05 Public access to information.

(a) A copy of a rule proposed under this part, and material supporting the rule, will be available for public inspection during normal business hours at the EPA Regional Office proposing the rulemaking action and at EPA's Public Information Reference Unit at 401 M Street SW., Washington, D.C. 20460. This material will be available beginning with the date notice is published in the **FEDERAL REGISTER** in accordance with § 65.04 (a)(1) or (b)(1).

(b) The EPA Regional Office will provide reasonable public access during normal business hours to any written comments submitted in response to a notice published under this section and to the record of any public hearing held under § 65.04(a).

§ 65.06 Effective date of federally promulgated or approved delayed compliance orders and revisions thereto.

(a) Federal delayed compliance orders and revisions to Federal delayed compliance orders promulgated in this part become effective under section 113(d) of the Act upon publication in the **FEDERAL REGISTER** of the notice described in § 65.04(a)(6).

(b) EPA approval of State delayed compliance orders issued to major stationary sources and revisions to such orders approved under this part become effective under section 113(d) of the Act upon publication in the **FEDERAL REGISTER** of the notice described in § 65.04(b)(4).

(c) A delayed compliance order issued by a State to a source other than a major stationary source and any revision to such an order becomes effective under section 113(d) of the Act upon final State action, unless the Administrator disapproves the order

or revision in accordance with § 65.04(b).

§ 65.07 Submittal of State delayed compliance orders to the Administrator.

In order to obtain EPA approval of a State delayed compliance order under section 113(d)(2) of the Act, the State shall transmit a copy of the order and a request for EPA action to the appropriate EPA Regional Office, to the attention of the Director, Enforcement Division. In order to obtain EPA approval of a delayed compliance order issued by a local agency, the order must be submitted to EPA by the local agency either through the appropriate State Air Pollution Control Agency or in accordance with procedures established by the State which allow direct submittal of the order to EPA by the local agency. The EPA Regional Offices and addresses are identified at 40 CFR 52.16.

§ 65.08 Severability of provisions.

The provisions promulgated in this part and the various applications thereof are distinct and severable. If any provision of this part or the application thereof to any person or circumstances is held invalid, such invalidity shall not affect other provisions, or the application of such provisions to other persons or circumstances, which can be given effect without the invalid provision of application.

§ 65.09 Failure to comply with federally promulgated or approved delayed compliance orders.

(a) If the Administrator determines that a stationary source is in violation of a requirement contained in an order promulgated or approved under this part, he shall, as provided by section 113(d)(9) of the Act:

(1) Enforce such requirement under section 113 (a), (b), or (c) of the Act;

(2) Revoke the order in accordance with § 65.04(d) and enforce compliance with the State implementation plan provisions(s) covered by the order;

(3) Give notice of noncompliance and commence action under section 120 of the Act; or

(4) Take any appropriate combinations of these actions.

Under section 304 of the Act, and in accordance with 40 CFR Part 54, any person may commence a civil action against an owner or operator of a source which is alleged to be in violation of any order promulgated or approved under this part.

§ 65.10 Termination of delayed compliance orders.

(a) Delayed compliance orders issued or approved by EPA under this part may be terminated if the Administrator determines in accordance with section 113(d)(8) of the Act that the inability of the stationary source to comply with the State implementation plan provision(s) covered by the order no longer exists.

(b) The duration of any provision in a delayed compliance order which is imposed under authority other than section 113(d) of the Act is governed by the terms of that provision.

Subparts B-C—[Reserved]

Subpart D—Arizona

§ 65.70 Federal delayed compliance orders issued under section 113(d) (1), (3), (4) and (5) of the Act.

The delayed compliance orders referenced below have been issued by the Administrator in accordance with section 113(d) of the Act and with this part. With regard to each order, the Administrator has made all the determinations and findings which are necessary for issuance of the order under section 113(d) of the Act.

Source	Location	Order No.	SIP regulation(s) involved	Date of FEDERAL REGISTER promulgation	Final compliance date
Phelps Dodge Co	Ajo, Ariz	9-81-27	4) CFR 51.125(d) 40 CFR 52.126(b)	Jan. 12, 1982	Dec. 31, 1985.

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Source	Location	Order No.	SIP regulation(s) involved	Date of FEDERAL REGISTER promulgation	Final compliance date
Phelps Dodge Co	Morenci, Ariz	9-81-28	40 CFR 51.125(d) Reg 7-1-3.6 (Ariz. Dept. of Health)	do	Jan. 1, 1985

[43 FR 44523, Sept. 28, 1978, as amended at 47 FR 1294, Jan. 12, 1982; 48 FR 44745, Sept. 28, 1983]

§§ 65.71—65.72 [Reserved]

Subparts E—J—[Reserved]

Subpart K—Florida

§ 65.140 [Reserved]

§ 65.141 EPA approval of State delayed compliance orders issued to major stationary sources.

The State orders identified below have been approved by the Adminis-

trator in accordance with section 113(d)(2) of the Act and with this part. With regard to each order, the Administrator has made all the determinations and findings which are necessary for approval of the order under section 113(d) of the Act.

Source	Location	Order Number	SIP regulation(s) involved	Date of FEDERAL REGISTER proposal	Final compliance date
National Can Co	Tarrant, Alabama	DCO-IV-8406	Jefferson Co. Subparagraphs 8.11.1(c)(1) and 8.11.1(c)(4) AL Dept. of Environmental Mgmt. Subparagraphs 6.11.1(c)(1) and 6.11.1(c)(4)	Oct. 9, 1984	Dec. 31, 1985
Continental Can Co., Inc	Tampa, FL	DCO-IV-8405	Rule 17- 2.650(1)(f)1. and Rule 17- 2.650(1)(b)1.d.(ii)	Oct. 9, 1984	Sept. 1, 1985

[43 FR 44523, Sept. 28, 1978, as amended at 48 FR 44745, Sept. 29, 1983; 50 FR 3340 and 3341, Jan. 24, 1985]

§ 65.142 [Reserved]

Subpart L—Georgia

§ 65.150 Federal delayed compliance orders issued under section 113(d) (1), (3), (4) and (5) of the Act.

The delayed compliance orders referenced below have been issued by the

Administrator in accordance with section 113(d) of the Act and with this part. With regard to each order, the Administrator has made all the determinations and findings which are necessary for issuance of the order under section 113(d) of the Act.

Source	Location	Order Number	SIP regulation(s) involved	Date of FEDERAL REGISTER promulgation	Final compliance date
Atlanta Packaging	Atlanta, GA	EPD-AQC-255	Chapter 391-3-1, rule .02(2)(mm)	May 29, 1985	Dec. 1, 1986.
General Motors	Doraville, GA	EPD-AQC-256	Chapter 391-3-1, rule .02(2)(ii)	May 29, 1985	Oct. 31, 1985.
International Paper Co	Atlanta, GA	EPD-AQC-257	Chapter 391-3-1, rule .02(2)(mm)	May 29, 1985	Dec. 1, 1986.
Mead Packaging	Atlanta, GA	EPD-AQC-258	Chapter 391-3-1, rule .02(2)(mm)	May 29, 1985	Mar. 1, 1985.
Purpack	Atlanta, GA	EPA-AQC-259	Chapter 391-3-1, rule .02(2)(mm)	May 29, 1985	Dec. 1, 1986.

[43 FR 44523, Sept. 28, 1978, as amended at 48 FR 44745, Sept. 29, 1983; 50 FR 48582-48586, Nov. 26, 1985]

§§ 65.151-65.152 [Reserved]

Subparts M-N-[Reserved]

Subpart O-Illinois

§ 65.180 Federal delayed compliance orders issued under section 113(d) (1) (3), (4) and (5) of the Act.

The delayed compliance orders referenced below have been issued by the

Administrator in accordance with section 113(d) of the Act and with this part. With regard to each order, the Administrator has made all the determinations and findings which are necessary for issuance of the order under section 113(d) of the Act.

Source	Location	Order Number	SIP regulation(s) involved	Date of FEDERAL REGISTER proposal	Final compliance date
Engineered coated products	Northbrook, IL	EPA-5-84-A-	205(n)	Apr. 16, 1984	Dec. 1, 1984

[43 FR 44523, Sept. 28, 1978, as amended at 48 FR 44745, Sept. 29, 1983; 49 FR 28560, July 13, 1984]

§§ 65.181-65.182 [Reserved]

Subparts P-S-[Reserved]

Subpart T-Louisiana

§ 65.230 [Reserved]

§ 65.231 EPA approval of State delayed compliance orders issued to major stationary sources.

The State orders identified below have been approved by the Adminis-

trator in accordance with section 113(d)(2) of the Act and with this part. With regard to each order, the Administrator has made all the determinations and findings which are necessary for approval of the order under section 113(d) of the Act.

Source	Location	Order Number	SIP regulation(s) involved	Date of FEDERAL REGISTER promulgation	Final compliance date
Conoco Incorporated	Westlake, LA	None	§ 22.3	Sept. 6, 1983	Dec. 31, 1983

Source	Location	Order Number	SIP regulation(s) involved	Date of FEDERAL REGISTER promulgation	Final compliance date
Formosa Plastics Corp	Baton Rouge, LA	do	§ 22.3 & 22.8	do	Do.
Vulcan Materials Co	Geismar, LA	do	§ 22.8	do	Dec. 31, 1984

[43 FR 44523, Sept. 28, 1978, as amended at 48 FR 44745, Sept. 29, 1983; 48 FR 55286, Dec. 12, 1983]

§ 65.232 [Reserved]

Subpart U—[Reserved]**Subpart V—Maryland**

§ 65.250 Federal delayed compliance orders issued under section 113(d) (1), (3), (4) and (5) of the Act.

The delayed compliance orders referenced below have been issued by the

Administrator in accordance with section 113(d) of the Act and with this part. With regard to each order, the Administrator has made all the determinations and findings which are necessary for issuance of the order under section 113(d) of the Act.

Source	Location	Order No.	SIP regulation(s) involved	Date of FEDERAL REGISTER promulgation	Final compliance date
Naval Ordnance Station, Goddard Units 1-3	Indian Head, Md	R-III-CC-007	COMAR § 10.03, 40.02B, 103.03, 40.02D, 10.03, 40.02C(2)	June 23, 1983.	Mar. 1, 1985.

[43 FR 44523, Sept. 28, 1978, as amended at 48 FR 44745, Sept. 29, 1983]

§§ 65.251—65.252 [Reserved]

Subpart W—Massachusetts

§ 65.260 Federal delayed compliance orders issued under section 113(d) (1), (3), (4) and (5) of the Act.

The delayed compliance orders referenced below have been issued by the

Administrator in accordance with section 113(d) of the Act and with this part. With regard to each order, the Administrator has made all the determinations and findings which are necessary for issuance of the order under section 113(d) of the Act.

Source	Location	Order No.	SIP regulation(s) involved	Date of FEDERAL REGISTER promulgation	Final compliance date
Holyoke Water Power Mt. Tom Unit.	Holyoke, Mass.....		310 CMR 7.02(B), 7.05(4) and 7.06(1).	Nov. 24, 1981.....	Feb. 15, 1984.....
New England Power Co., Salem Harbor Units 1, 2 and 3.	Salem, Mass.....		310 CMR 7.02(B), 7.05(4) and 7.06.	Feb. 9, 1982.....	No later than Dec. 31, 1982.....
Montaup Electric Co., Somerset Station.	Somerset, Mass.....		310 CMR 7.02(B) and 7.06(1).	Mar. 22, 1983.....	Do.

[43 FR 44523, Sept. 28, 1978, as amended at 48 FR 44746, Sept. 29, 1983]

§§ 65.261—65.262 [Reserved]

Subpart X—Michigan

§ 65.270 Federal delayed compliance orders issued under section 113(d) (1), (3), (4) and (5) of the Act.

The delayed compliance orders referenced below have been issued by the

Administrator in accordance with section 113(d) of the Act and with this part. With regard to each order, the Administrator has made all the determinations and findings which are necessary for issuance of the order under section 113(d) of the Act.

Source	Location	Order Number	SIP regulation(s) involved	Date of FEDERAL REGISTER proposal	Final compliance date
General Motors Corp., Detroit Diesel Allison-Redford Plant	Detroit, MI	To be assigned	R336.1621	May 9, 1985	12/1/84

[43 FR 44523, Sept. 28, 1978, as amended at 48 FR 44745, Sept. 29, 1983; 50 FR 32413, Aug. 12, 1985]

§ 65.271 EPA approval of State delayed compliance orders issued to major stationary sources.

The State orders identified below have been approved by the Administrator in accordance with section

113(d)(2) of the Act and with this part. With regard to each order, the Administrator has made all the determinations and findings which are necessary for approval of the order under section 113(d) of the Act.

Source	Location	Order Number	SIP regulation(s) involved	Date of FEDERAL REGISTER proposal	Final compliance date
Barker and Sons Finishing Co., Inc.	Oshtemo, MI	To be Assigned	Code 1980 AACCS R336.1621	Sept. 25, 1983	June 30, 1985
Chrysler Corp., Warren Truck Plant	Warren, MI		Sept. 25, 1984	Code 1980 AACCS R336.1331	Jan. 31, 1985
General Motors Corporation Saginaw Division	Saginaw, MI	EPA-5-86-113(d)-28	Code 1980 AACCS R336.1621	May 2, 1986	Dec. 31, 1986
General Motors Corp., Truck and Bus Group	Pontiac, MI	To be assigned	R336.1621	June 20, 1986	Dec. 31, 1986
General Motors Corporation, Truck and Bus Group, Pontiac West Assembly Plant	Pontiac, MI	To be assigned	Code 1980 AACCS R336.1610	Jan. 29, 1987	Aug. 1, 1987

[43 FR 44523, Sept. 28, 1978, as amended at 49 FR 49463 and 49464, Dec. 20, 1984; 51 FR 33266, Sept. 19, 1986; 52 FR 3800, Feb. 6, 1987; 52 FR 16248, May 4, 1987]

§ 65.272 EPA disapproval of State delayed compliance orders.

The State orders identified below have been disapproved by the Administrator in accordance with section

113(d)(2) of the Act and with this part. With regard to each order, the Administrator has determined that it does not satisfy the applicable requirements of section 113(d) of the Act.

Source	Location	Order Number	SIP regulation(s) involved	Date of FEDERAL REGISTER proposal	Final compliance date
Riverside correctional facility	Ionia, MI		Code 1980 AACS, R336.1331	Apr. 18, 1984	

[43 FR 44523, Sept. 28, 1978, as amended at 49 FR 33129, Aug. 21, 1984]

Subparts Y-Z—[Reserved]

Subpart AA—Missouri

§ 65.300 [Reserved]

§ 65.301 EPA approval of State delayed compliance orders issued to major stationary sources.

The State orders identified below have been approved by the Adminis-

trator in accordance with section 113(d)(2) of the Act and with this part. With regard to each order, the Administrator has made all the determinations and findings which are necessary for approval of the order under section 113(d) of the Act.

Source	Location	Order Number	SIP regulation(s) involved	Date of FEDERAL REGISTER promulgation	Final compliance date
American Can Co.	St. Louis, MO	DCO-V11-026	10CSR10-5.330	Jan. 7, 1987	Dec. 31, 1986 Nov. 15, 1987

[48 FR 44745, Sept. 29, 1983, as amended at 52 FR 17760, May 12, 1987]

§ 65.302 [Reserved]

Subparts BB-EE—[Reserved]

Subpart FF—New Jersey

§ 65.350 Federal delayed compliance orders issued under section 113(d) (1), (3), (4) and (5) of the Act.

The delayed compliance orders referenced below have been issued by the

Administrator in accordance with section 113(d) of the Act and with this part. With regard to each order, the Administrator has made all the determinations and findings which are necessary for issuance of the order under section 113(d) of the Act.

Source	Location	Order No.	SIP regulation(s) involved	Date of FEDERAL REGISTER promulgation	Final compliance date
Atlantic City Electric Deepwater Unit B	Pennsville, N.J.	00109	N.J.A.C. 7.27-3.1 et seq. and 7.27-4.1 et seq. and 7.27-8.1 et seq.	Jan. 26, 1981	Oct. 1, 1983

[43 FR 44523, Sept. 28, 1978, as amended at 48 FR 44745, Sept. 29, 1983]

Environmental Protection Agency

§ 65.431

§§ 65.351—65.352 [Reserved]

Subparts GG—MM—[Reserved]

Subpart NN—Pennsylvania

§ 65.430 [Reserved]

§ 65.431 EPA approval of State delayed compliance orders issued to major stationary sources.

The State orders identified below have been approved by the Adminis-

trator in accordance with section 113(d)(2) of the Act and with this part. With regard to each order, the Administrator has made all the determinations and findings which are necessary for approval of the order under section 113(d) of the Act.

Source	Location	Order No.	Dated of FR proposal	SIP regulation involved	Final compliance date
ACF Industries, Inc.	Milton Township, Northumberland County, PA		Feb. 26, 1986	Section 129.52 Title 25 of the PA Code	Apr. 21, 1987
Allegheny Label, Inc.	Cheswick Township, Allegheny County, PA		Aug. 21, 1986	Section 531(A) of Allegheny County Health Department, Rules & Regulations, Article XX, Air Pollution Control	Apr. 21, 1987
Allied Paper Division of SCM Corporation	Phoenixville, PA		Aug. 22, 1984	Section 129.52 of Title 25	Apr. 9, 1985
Anchor Hocking Corporation	Connellsville, PA		Sept. 25, 1984	Section 129.52 of Title 25	Apr. 21, 1987
Apollo Metals, Inc.	Bethlehem, PA		Aug. 22, 1984	Section 129.52 of Title 25	Apr. 9, 1985
Boyetown Packaging Corp.	Colebrookdale Township, Berks County, PA			Section 129.52 and Section 129.67 of Title 25	Apr. 21, 1987
Brown Group Recreational Products, Inc., Hadelrom Division	Bedford, PA		Dec. 5, 1984	Section 129.52 of Title 25	Apr. 21, 1985
Cole Division Joyce International	Spring Garden and Springettsbury, PA		Aug. 22, 1984	Section 129.52 of Title 25	Apr. 9, 1985
Container Corporation of America	Upper Providence Township, Montgomery County		Mar. 26, 1985	Section 129.67 of Title 25	Dec. 31, 1985
Fisher Scientific Company	Indiana Township, Indiana County, PA		Apr. 21, 1986	Section 129.52, Table 1, Miscellaneous Metal Parts and Products, 10(f), Title 25 Pa. Code	Apr. 21, 1987
Fichtlung Coated Products, Division of Litton Business Systems, Inc.	Moosic, PA		Oct. 2, 1984	Section 129.52 of Title 25	Apr. 9, 1985
Franklin Steel Co.	Franklin, PA		Aug. 22, 1984	Section 129.52 of Title 25	Sept. 30, 1985
Keystone Lamp, Subsidiary of Kiddle, Inc.	Washington Township, PA		Aug. 22, 1985	Section 129.52 of Title 25	Apr. 9, 1985
National Can Corporation	Upper Macungie, Pennsylvania		Feb. 7, 1985	Section 129.52 of Title 25	Apr. 9, 1985

Source	Location	Order No.	Dated of FR proposal	SIP regulation involved	Final compliance date
Papercraft Corporation	Papercraft Park, Allegheny County, PA		Aug. 21, 1986	Section 531(A) of Allegheny County Health Department, Rules & Regulations, Article XX, Air Pollution Control	Apr. 21, 1987
Philadelphia Textile Finishers, Inc.	Norristown, PA		Sept. 25, 1984	Section 129.52 of Title 25	Apr. 9, 1985
Reading Brdy Work, Inc.	Reading, Berks County, PA		May 28, 1986	Section 129.52 of Title 25	Apr. 21, 1987
Superpac, Inc.	Southampton, PA		Aug. 22, 1984	Section 129.67 of Title 25	June 30, 1985
Victor Balata Belting Co.	Wilson Borough, PA		Aug. 22, 1984	Section 129.52 of Title 25	Apr. 9, 1985
Wyomissing Converting Co.	Berks County, PA			Section 129.67 of Title 25	Oct. 1, 1986
Zapata Industries	West Mahoney Twp. Schuylkill County, PA		Feb. 6, 1986	Section 129.52 of Title 25	Apr. 21, 1987

[43 FR 44523, Feb. 15, 1978, as amended at 48 FR 44745, Sept. 29, 1983; 50 FR 772-775, Jan. 7, 1985; 50 FR 5251-5253, Feb. 7, 1985; 50 FR 5582, Feb. 11, 1985; 50 FR 13971, Apr. 9, 1985; 50 FR 20760, May 20, 1985; 50 FR 24197, June 10, 1985; 51 FR 9957, Mar. 24, 1986; 51 FR 25695, July 16, 1986; 51 FR 26892, July 28, 1986; 51 FR 28225, Aug. 6, 1986; 51 FR 30094, Aug. 22, 1986; 51 FR 33267, Sept. 19, 1986; 51 FR 36692, Oct. 15, 1986; 52 FR 7864, 7865, Mar. 13, 1987]

§ 65.432 [Reserved]

Subparts OO-QQ—[Reserved]

Subpart RR—Tennessee

§ 65.470 [Reserved]

§ 65.471 EPA approval of State delayed compliance orders issued to major stationary sources.

The State orders identified below have been approved by the Adminis-

trator in accordance with section 113(d)(2) of the Act and with this part. With regard to each order, the Administrator has made all the determinations and findings which are necessary for approval of the order under section 113(d) of the Act.

Source	Location	Order No.	SIP regulation(s) involved	Date of FR proposal	Final compliance date
Bryce Corp	Memphis	84-0321-0420-1	MCC—§ 3-22, Ref TAQA, 1200-3-18.06, 1200-3-18.29	Sept. 20, 1984	Dec. 31, 1985
Cleo Wrap	Memphis	84-0153-01	MCC, 322 Ref TAQA 1200-3-18-29	Oct. 9, 1984	Dec. 31, 1985
Jehi Cooperage	Memphis	85-0154-01	MCC § 3-22 Ref TAQA 1200-3-18-21	Sept. 18, 1985	Dec. 31, 1985
Sandusky-Memphis Metal Cabinets, Inc.	Millington, Tennessee	DCO/IV/8403	1200-3-18.15	Oct. 3, 1984	Nov. 1, 1984

Environmental Protection Agency

§ 65.510

[43 FR 44523, Sept. 28, 1978, as amended at 48 FR 44745, Sept. 29, 1983; 50 FR 935, Jan. 8, 1985; 50 FR 1852, Jan. 14, 1985; 50 FR 4512, Jan. 31, 1985; 50 FR 48585, Nov. 26, 1985]

§ 65.472 [Reserved]

Subpart SS—Texas

§ 65.480 [Reserved]

§ 65.481 EPA approval of State delayed compliance orders issued to major stationary sources.

The State orders identified below have been approved by the Adminis-

trator in accordance with section 113(d)(2) of the Act and with this part. With regard to each order, the Administrator has made all the determinations and findings which are necessary for approval of the order under section 113(d) of the Act.

Source	Location	Order Number	SIP regulation(s) involved	Date of FEDERAL REGISTER proposal	Final compliance date
Arrow Industries, Inc.	Carrollton, TX	TACB No. 85-10	§ 115.201	1/7/86	12/31/85
Arrow Industries, Inc.	Farmers Branch, TX	TACB No. 85-11	§ 115.201	1/7/86	12/31/85
Deco Inc.	Dallas, TX	TACB No. 84-12	Rule 115.201	5/16/85	12/31/85
Proton Pkg. Inc.	Dallas, TX	TACB No. 84-14	Rule 115.201	5/16/85	12/31/85
Pimpack Inc. (*)	Grand Prairie, TX	TACB No. 84-10	Rule 115.201	5/16/85	12/31/85

* Section 120 noncompliance penalty language in TACB No. 84-10 may be misleading. Source is subject to noncompliance penalties after December 31, 1982, unless exempted under section 120(a)(2) (B) or (C).

[43 FR 44523, Sept. 28, 1978, as amended at 50 FR 37179—37181, Sept. 12, 1985; 51 FR 24657, July 8, 1986]

§ 65.482 [Reserved]

Subparts TT-UU—[Reserved]

Subpart VV—Virginia

§ 65.510 Federal delayed compliance orders issued under section 113(d) (1), (3), (4) and (5) of the Act.

The delayed compliance orders referenced below have been issued by the

Administrator in accordance with section 113(d) of the Act and with this part. With regard to each order, the Administrator has made all the determinations and findings which are necessary for issuance of the order under section 113(d) of the Act.

Source	Location	Order No.	SIP regulation(s) involved	Date of FEDERAL REGISTER promulgation	Final compliance date
Virginia Electric Power Co. Possum Pt. Units 2 & 3	Possum Point, VA	R-111-CC-005	VAPCB Section IV, Rules 2 & 3	Mar. 9, 1981	Apr. 1, 1984, Nov. 1, 1982

[43 FR 44523, Sept. 28, 1978, as amended at 48 FR 44746, Sept. 29, 1983]

§ 65.521**40 CFR Ch. I (7-1-90 Edition)****§§ 65.511-65.512 [Reserved]****Subpart WW—Washington****§ 65.520 [Reserved]****§ 65.521 EPA approval of State delayed compliance orders issued to major stationary sources.**

The State orders identified below have been approved by the Adminis-

trator in accordance with section 113(d)(2) of the Act and with this part. With regard to each order, the Administrator has made all the determinations and findings which are necessary for approval of the order under section 113(d) of the Act.

Source	Location	Order No.	SIP regulation(s) involved	Date of FEDERAL REGISTER promulgation	Final compliance date
Fletcher Oil Co	Takoma, Wash	W-13	WAC 173-490-040 WAC 173-490-201 PSAPCA 2	May 19, 1982	Jan. 1, 1983

[43 FR 44523, Sept. 28, 1978]

EDITORIAL NOTE: For FEDERAL REGISTER citations affecting § 65.521, see the List of CFR Sections Affected appearing in the Finding Aids section of this volume.

§ 65.522 [Reserved]**Subpart XX—[Reserved]****Subpart YY—Wisconsin****§ 65.540 Federal delayed compliance orders issued under section 113(d) (1), (3), (4) and (5) of the Act.**

The delayed compliance orders referenced below have been issued by the

Administrator in accordance with section 113(d) of the Act and with this part. With regard to each order, the Administrator has made all the determinations and findings which are necessary for issuance of the order under section 113(d) of the Act.

Source	Location	Order No.	SIP regulation(s) involved	Date of FR proposal	Final compliance date
C.B. Henschel Manufacturing Co., Inc	New Berlin, WI	EPA-84-CAA-113-V-01	NR 154.13(4)(e), NR 154.13(12)(b)	July 11, 1984	Sept. 15, 1984

[43 FR 44523, Sept. 28, 1978, as amended at 48 FR 44745, Sept. 29, 1983; 49 FR 43648, Oct. 31, 1984]

§§ 65.541—65.542 [Reserved]

Subpart ZZ—[Reserved]

Subpart AAA—Guam

§ 65.560 Federal delayed compliance orders issued under section 113(d) (1), (3), (4) and (5) of the Act.

The delayed compliance orders referenced below have been issued by the

Administrator in accordance with section 113(d) of the Act and with this part. With regard to each order, the Administrator has made all the determinations and findings which are necessary for issuance of the order under section 113(d) of the Act.

Source	Location	Order No.	SIP regulation(s) involved	Date of FEDERAL REGISTER promulgation	Final compliance date
Guam Power Authority	Agana, Guam	9-80-8	Section 13.4 of Chapter 13, Guam Air Pollution Standards and Regulations	Apr. 27, 1981	Feb. 15, 1985

[43 FR 44523, Sept. 28, 1978, as amended at 48 FR 44746, Sept. 29, 1983]

§§ 65.561—65.562 [Reserved]

Subparts BBB—EEE—[Reserved]

PART 66—ASSESSMENT AND COLLECTION OF NONCOMPLIANCE PENALTIES BY EPA

Subpart A—Purpose and Scope

Sec.

- 66.1 Applicability and effective date.
- 66.2 Program description.
- 66.3 Definitions.
- 66.4 Limitation on review of regulations.
- 66.5 Savings clause.
- 66.6 Effect of litigation; time limits.

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- 66.11 Issuance of notices of noncompliance.
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- 66.33 De Minimis exemptions: malfunctions.
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Subpart E—Decisions on Exemption Requests and Challenges to Notices of Noncompliance

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- 66.43 Final decision; submission of penalty calculation.

Subpart F—Review of Penalty Calculation

- 66.51 Action upon receipt of penalty calculation.
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Subpart G—Payment

- 66.61 Duty to pay.
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Subpart H—Compliance and Final Adjustment

- 66.71 Determination of compliance.
- 66.72 Additional payment or reimbursement.
- 66.73 Petition for reconsideration and procedure for hearing.
- 66.74 Payment or reimbursement.

Subpart I—Final Action

- 66.81 Final action.

Subpart J—Supplemental Rules for Formal Adjudicatory Hearings

- 66.91 Applicability of supplemental rules.
- 66.92 Commencement of hearings.
- 66.93 Time limits.
- 66.94 Presentation of evidence.
- 66.95 Decisions of Presiding Officer; appeal to the Administrator.

APPENDIX A—TECHNICAL SUPPORT DOCUMENT [NOTE]

APPENDIX B—INSTRUCTION MANUAL [NOTE]

APPENDIX C—COMPUTER PROGRAM [NOTE]

AUTHORITY: Sec. 120, Clean Air Act, as amended, 42 U.S.C. 7420.

SOURCE: 45 FR 50110, July 28, 1980, unless otherwise noted.

Subpart A—Purpose and Scope

§ 66.1 Applicability and effective date.

(a) This part applies to all proceedings for the assessment by EPA of a noncompliance penalty as provided by section 120 of the Clean Air Act. This penalty is designed to recover the economic advantage which might otherwise accrue to a source by reason of its failure to comply with air pollution control standards after receipt of a notice of noncompliance.

(b) These regulations shall be effective October 27, 1980.

§ 66.2 Program description.

This part sets forth the procedures by which EPA will administer the noncompliance penalty provisions of Section 120 of the Clean Air Act. Subpart A describes the scope of the part, defines key terms and states the manner of operation of these provisions. Subpart B states which sources of air pollution are subject to these penalties and the form and substance of the notice of noncompliance. Subpart C and the accompanying Technical Support Document and Manual state how

a source must compute the penalty which it owes. Subpart D describes the conditions under which an exemption from the penalty may be available, and Subpart E sets forth the procedures for requesting such an exemption. Subpart F states how EPA will review penalties calculated by sources under Subpart C, and Subpart G describes the method of payment. Subpart H provides for adjustment of the penalty after the source has come into compliance and the actual costs of doing so are known. Finally, Subpart I states which actions under these regulations are subject to judicial review and on what conditions, and Subpart J provides supplemental procedures for adjudicatory hearings.

§ 66.3 Definitions.

In this part and Part 67:

(a) "Act" means the Clean Air Act, 42 U.S.C. 7401 *et seq.* as amended on August 7, 1977, except where the context specifically indicates otherwise.

(b) "Affiliated entity" means a person who directly, or indirectly through one or more intermediaries, controls, is controlled by, or is under common control with the owner or operator of a source.

(c) "Applicable legal requirements" means any of the following:

(1) In the case of any major source, any emission limitation, emission standard, or compliance schedule under any EPA-approved State implementation plan (regardless of whether the source is subject to a Federal or State consent decree);

(2) In the case of any source, an emission limitation, emission standard, standard of performance, or other requirement (including, but not limited to, work practice standards) established under section 111 or 112 of the Act;

(3) In the case of a source that is subject to a federal or federally approved state judicial consent decree or EPA approved extension, order, or suspension, any interim emission control requirement or schedule of compliance under that consent decree, extension, order or suspension;

(4) In the case of a nonferrous smelter which has received a primary non-

ferrous smelter order issued or approved by EPA under Section 119 of the Act, any interim emission control requirement (including a requirement relating to the use of supplemental or intermittent controls) or schedule of compliance under that order.

(d) "Approved Section 120 program" means a State program to assess and collect Section 120 penalties that has been approved by the Administrator.

(e) "Computer program" means the computer program used to calculate noncompliance penalties under section 120 of the Clean Air Act. This computer program appears as Appendix C to these regulations.

(f) "Control" (including the terms "controlling", "controlled by", and "under common control with") means the power to direct or cause the direction of the management and policies of a person or organization, whether by the ownership of stock, voting rights, by contract, or otherwise.

(g) "Major stationary source" means any stationary facility or source of air pollutants which directly emits, or has the potential to emit, one hundred tons per year or more of any air pollutant regulated by EPA under the Clean Air Act.

(h) "Manual" means the "Noncompliance Penalties Instruction Manual" which accompanies these regulations. This Manual appears as Appendix B to these regulations.

(i) "Owner or operator" means any person who owns, leases, operates or supervises a facility, building, structure or installation which emits or has the potential to emit any air pollutant regulated by EPA under the Act.

(j) "Potential to emit" means the capability at maximum design capacity to emit a pollutant after the application of air pollution control equipment. Annual potential shall be based on the larger of the maximum annual rated capacity of the stationary source assuming continuous operation, or on a projection of actual annual emissions. Enforceable permit conditions on the type of materials combusted or processed may be used in determining the annual potential. Fugitive emissions, to the extent quantifiable, will be considered in determining annual potential for those stationary sources

whose fugitive emissions are regulated by the applicable state implementation plan.

(k) "Source" means any source of air pollution subject to applicable legal requirements as defined in paragraph (c).

(l) "Technical Support Document" means the "Noncompliance Penalties Technical Support Document" which accompanies these regulations. The Technical Support Document appears as Appendix A to these regulations.

All other terms are defined as they are in the Act.

§ 66.4 Limitation on review of regulations.

No applicable legal requirement, which could have been reviewed or challenged by means of the timely filing of an appropriate petition, no provision of this part or Part 67 or Appendices A, B or C, may be challenged, reviewed or re-examined in any hearing conducted under this part or Part 67. This limitation on review includes, but is not limited to:

(a) Arguments that the statute is more or less restrictive than the regulations, e.g., that exemptions other than those provided herein should be granted.

(b) Arguments that the economic model does not accurately calculate the economic benefits of noncompliance, or that parameters, terms and conditions other than those provided for in the model should be used or that evidence other than that described in the Technical Support Document for establishing inputs should be considered.

§ 66.5 Savings clause.

Proceedings under these regulations for imposition of a penalty under Section 120 are in addition to any other proceedings related to permits, orders, payments, sanctions or other requirements or State of Federal law. No action under this part or Part 67 shall affect in any way any administrative, civil or criminal enforcement proceeding brought under any provision of the Clean Air Act or State or local law.

§ 66.6 Effect of litigation; time limits.

(a) The existence of any litigation on the validity of these regulations shall not affect the authority of the Agency to issue notices of noncompliance or to conduct subsequent administrative proceedings under Parts 66 and 67.

(b) Failure of the Administrator or the Presiding Officer at a hearing to meet any of the time limits contained in Parts 66 and 67 shall not affect the validity of any proceeding under these regulations.

(c) The filing of any petition for reconsideration under this part or Part 67 or the institution of EPA review of a State determination under Part 67 shall not toll the accrual of noncompliance penalties. The penalty will be calculated from the date on which the source owner or operator receives a notice of noncompliance.

Subpart B—Notice of Noncompliance

§ 66.11 Issuance of notices of noncompliance.

(a) The Administrator shall issue a notice of noncompliance to the owner or operator of any source which he determines is in violation of applicable legal requirements and which is located in a State without an approved Section 120 program.

(b) The Administrator shall send a notice of noncompliance to the owner or operator of any source located in a State with an approved Section 120 program when he determines as provided in Part 67 that the source is in violation of applicable legal requirements and the State has failed to send a notice of noncompliance to it, or has failed to pursue diligently any subsequent steps for the assessment or collection of the penalty.

(c) Failure of EPA or a State to issue a notice of noncompliance within 30 days after discovery of a violation shall not affect the obligation of a source owner or operator to pay a noncompliance penalty but shall affect the date from which the penalty is calculated. The penalty shall be calculated from the earliest date that the owner or operator of the source received a notice of noncompliance

under this section, whether issued by EPA or the State.

§ 66.12 Content of notices of noncompliance.

(a) Each notice of noncompliance shall be in writing and shall include:

(1) A specific reference to each applicable legal requirement of which the source is in violation;

(2) A brief statement of the factual basis for the finding of violation, together with a reference to any supporting materials and a statement of when and where they may be inspected;

(3) Instructions on calculating the amount of the penalty owed and the schedule for payments. Such instructions shall include (i) a statement of the date from which penalties should be calculated and (ii) a copy of the Technical Support Document and the Manual;

(4) Notice of the right to petition for a hearing to challenge the finding of noncompliance or to claim an exemption; and

(5) Notice that the penalty continues to accrue during the pendency of any hearings granted under this part or Part 67.

(b) Each notice of noncompliance shall be transmitted to the source owner or operator either by personal service or by registered or certified mail, return receipt requested.

§ 66.13 Duties of source owner or operator upon receipt of a notice of noncompliance.

(a) Within forty-five days after receiving a notice of noncompliance a source owner or operator shall either:

(1) Calculate the amount of the penalty owed and the appropriate quarterly payment schedule, as provided in the Technical Support Document and Instruction Manual, and transmit that calculation, together with supporting data sufficient to allow verification of the penalty calculation, to the Administrator; or

(2) Submit a petition for reconsideration, alleging that the source is not in violation of applicable legal requirements or that the source owner or operator is entitled to an exemption pur-

suant to §§ 66.31 through 66.33, or both. A source owner or operator must present both grounds in the petition if he wishes to preserve a claim to an exemption in the event that the source is found to be in violation. Issues relating to the existence of a violation or entitlement to an exemption not raised in the petition shall be deemed waived.

(b) Any submittal pursuant to this subsection shall specify the identity of the person responsible for the payment of any noncompliance penalty, and to whom any reimbursement, if necessary, shall be sent.

(c) A source owner or operator may amend any petition for reconsideration pursuant to paragraph (a) of this section within 45 days from receipt of a notice of noncompliance. Amendment of such petition after 45 days will be permitted only if based on unforeseeable conditions occurring after termination of the 45 day period, or upon consent of the Administrator.

Subpart C—Calculation of Noncompliance Penalties

§ 66.21 How to calculate the penalty.

(a) All noncompliance penalties shall be calculated in accordance with the Technical Support Document and the Manual.

(b) Where the Administrator determines that no existing technology or other emissions control method results in emission levels which satisfy the applicable legal requirement, the penalty calculation shall be based on the cost of the capital equipment, operation and maintenance practices, or other methods of control which best approximates the degree of control required. In such a case, the Administrator may include in the penalty the costs of participation in an EPA approved research and development program where he determines that such participation would be appropriate. Information on appropriate research and development programs will be available from the regional offices or from the Office of Research and Development.

§ 66.22 Contracting out penalty calculation.

Upon the failure of a source owner or operator, who does not submit a petition for reconsideration as provided in § 66.13(a)(2), to submit the information described in § 66.13(a)(1) within 45 days of receipt of a notice of noncompliance, or upon submission of incorrect information as determined pursuant to § 66.51, the Administrator may enter into a contract with any qualified person who is not an affiliated entity and who has no financial interest in the owner or operator of the source to assist in determining the amount of the penalty assessment or payment schedule with respect to such source owner or operator. The cost of this contract may be added to the penalty to be assessed against the owner or operator of the source. The data used in calculating the penalty shall be furnished to the source owner or operator at the time that the penalty calculation is reported.

§ 66.23 Interim recalculation of penalty.

(a) The Administrator, upon concluding that a previously approved penalty calculation no longer is accurate, may:

(1) Request, in writing, that the source owner or operator submit a revised calculation in the form specified in § 66.13(a). The Administrator shall respond to any information submitted in accordance with the provisions of § 66.51.

(2) Notify the source owner or operator, in writing, that the penalty has been recalculated based upon information in the Administrator's possession. The source owner or operator shall respond as provided in § 66.52.

(b) If a source owner or operator believes that, because of changed circumstances, a penalty calculation which has been accepted by EPA no longer is accurate, he may submit a revised penalty calculation and schedule to the Administrator. The revised calculation shall be in the form specified in § 66.13(a)(1). The Administrator shall respond in accordance with the provisions of § 66.51. The decision to accept the interim calculation or to grant a hearing on this issue shall be

solely within the discretion of the Administrator.

**Subpart D—Exemption Requests;
Revocation of Exemptions**

§ 66.31 Exemptions based on an order, extension or suspension.

(a) A source owner or operator who would otherwise be subject to a non-compliance penalty will be exempted from that penalty during the period for which, and upon a demonstration that, its noncompliance with applicable legal requirements is or was due solely to;

(1) A conversion by such source from the burning of petroleum products or natural gas, or both, as the permanent primary energy source to the burning of coal pursuant to an order under section 113(d)(5) or section 119 of the Act as in effect before August 7, 1977.

(2) In the case of a coal-burning source, the issuance of a prohibition to that source against burning petroleum products or natural gas, or both, by means of an order under sections 2(a) and (b) of the Energy Supply and Environmental Coordination Act of 1974, the Powerplant and Industrial Fuel Use Act, or under any legislation which amends or supersedes these provisions, *Provided*, That the source had received an extension under the second sentence of section 119(c)(1) of the Act as in effect before August 7, 1977.

(3) The use of innovative technology by the source owner or operator pursuant to an enforcement order under Section 113(d)(4) of the Act.

(4) An inability to comply with an applicable legal requirement resulting from reasons entirely beyond the control of the owner or operator of such source or of any affiliated entity, *Provided*, That

(i) The source owner or operator has received an order under section 113(d) (or an order under section 113 issued before August 7, 1977) or a federal or EPA-approved State judicial decree or order which has the effect of permitting a delay in complying with the legal requirement at issue, and

(ii) That the source owner or operator meets the requirements of paragraphs (c) and (d) of this section.

(5) The existence of an energy or employment emergency demonstrated by issuance of an order under section 110(f) or 110(g) of the Act, unless such order is disapproved by EPA.

(b) To qualify for an exemption under this section, the source owner or operator must have received the order, extension or suspension or consent decree described in the paragraph of the section pursuant to which the exemption is claimed. No exemption may be sought which, if granted, would exceed the terms of the relevant extension, order, suspension, or consent decree, except as provided in paragraph (e) of this section. No exemption may be sought which is based on a claim that the source owner or operator is entitled to any such order, extension, suspension, or consent decree even though it has not been issued.

(c) In any exemption claim based on paragraph (a)(4) of this section, the source owner or operator must demonstrate:

(1) That the source owner or operator or an affiliated entity in no manner sought, caused, encouraged or contributed to the inability; and

(2) That the source owner or operator in no way unduly delayed negotiation for needed equipment or fuel supply or made unusual demands not typical in its industry, or placed unusual restrictions on the supplier, or delayed in any other manner the delivery of goods or the completion of the necessary construction.

(d)(1) No exemption will be granted pursuant to paragraph (a)(4) of this section unless the owner or operator of the source demonstrates that, with respect to a situation described in paragraph (c), all reasonable steps were taken to prevent the situation causing the inability to comply, that procuring the needed pollution control equipment or fuel supply was given and continues to be given the highest possible priority in the planning and budgeting process of the owner or operator of the source, and that alternative sources of equipment and fuel have been explored without success.

(2) Any exemption granted under paragraph (a)(4) of this section shall cease to be effective when the inability to comply ceases to be entirely

beyond the control of the source owner or operator as defined in this section.

(e) Except in the case of exemptions based on orders under section 113 (d)(4) or (d)(5) or suspensions under section 110(g), the Administrator may grant an exemption with retroactive effect to the date of the event giving rise to the Section 120 predicate order, extension, suspension, or consent decree. In such cases, the exemption from the noncompliance penalty shall run from the date that the basis for the exemption first occurred.

(45 FR 50110, July 20, 1980, as amended at 50 FR 36734, Sept. 9, 1985)

§ 66.32 De Minimis exemptions.

(a) The Administrator may, upon notice and opportunity for public hearing, exempt the owner or operator of any source from a penalty where he finds that a particular instance of noncompliance was *de minimis* in nature and duration.

(b) A petition for an exemption on the ground that the violation described in a notice of noncompliance was *de minimis* in nature and duration may only raise issues related to entitlement to an exemption and shall contain or be accompanied by supporting documentation. Issues relating to entitlement to a *de minimis* exemption not raised in the petition shall be deemed waived.

(c) In ruling upon such a petition, the Administrator shall consider:

(1) The magnitude of the excess emissions and whether the source's noncompliance is recurring or persistent;

(2) The steps the source owner or operator is taking to eliminate the cause of the excess emissions and to minimize such emissions;

(3) Whether any significant economic savings are likely to accrue to the owner or operator of the source as a result of the noncompliance;

(4) The character of the emissions, and their impact on ambient air quality; and

(5) The duration of the violation.

(d) A hearing on a petition for a *de minimis* exemption shall be informal. The hearing shall be scheduled upon notice to the public. Reasonable op-

portunity to testify and for submission of questions by the public to the petitioner shall be afforded. The decision of the hearing officer will be made in writing within a reasonable period of time after the close of the hearing.

§ 66.33 De Minimis exemptions: malfunctions.

(a) The Administrator may, upon notice and opportunity for a public hearing, exempt the owner or operator of a source if he finds with respect to a particular instance of noncompliance, that such noncompliance was *de minimis* in nature and duration, and was caused solely by a sudden and unavoidable breakdown of process or pollution control equipment.

(b) A petition for an exemption on the ground that the violation was *de minimis* and was caused by a sudden and unavoidable breakdown of process or pollution control equipment may only raise issues related to entitlement to an exemption and shall contain or be accompanied by supporting documentation. Issues relating to entitlement to an exemption that are not raised in the petition shall be deemed waived. In making such finding the Administrator shall consider whether:

(1) The violation was *de minimis* in nature and duration within the meaning of § 66.32;

(2) The air pollution control equipment, process equipment, or processes, including appropriate back-up systems, were designed, and have been maintained and operated in a manner consistent with good practice for minimizing emissions;

(3) Repair of the malfunctioning equipment was undertaken and carried out in an expeditious fashion as soon as the owner or operator knew or should have known that the malfunction existed or that applicable emission limitations were being violated or were likely to be violated;

(4) All practicable steps were taken to minimize the impact of the excess emissions (including any bypass) on ambient air quality;

(5) The excess emissions were not part of a pattern indicative of inadequate design, operation, or maintenance;

(6) Off-shift and overtime labor were utilized where necessary to ensure that repairs were made as expeditiously as possible or that emissions were minimized to the maximum extent possible; and

(7) The level of economic savings, if any, accruing to the source owner or operator was *de minimis*.

(c) Any activity that could have been foreseen, avoided or planned for, or any breakdown that could have been avoided by the exercise of reasonable diligence shall not constitute grounds for an exemption under this section. Such activities include, but are not limited to, sudden breakdowns avoidable by better maintenance procedures, phasing in and out of process equipment and routine maintenance.

(d) A hearing on any petition for an exemption based upon the unavoidable breakdown of pollution control equipment shall be informal. The hearing shall be scheduled upon notice to the public. Reasonable opportunity to testify and for submission of questions by the public to the petitioner shall be afforded. The decision of the hearing officer will be made in writing within a reasonable period of time after the close of the hearing.

§ 66.34 Termination of exemptions.

(a) Exemptions pursuant to § 66.31(a) (1), (2), (3), and (5) will cease to be effective upon termination or revocation of the order on which the exemption is based.

(b) Exemptions pursuant to § 66.31(a)(4) will cease to be effective when the order or decree is terminated or revoked, or the inability to comply ceases to be for reasons entirely beyond the control of the source owner or operator as defined in § 66.31(c).

(c) Exemptions granted pursuant to § 66.32 or § 66.33 shall terminate at the time specified in the exemption.

(d) The Administrator shall notify the source owner or operator, in writing, that the exemption has terminated, and shall specify the date from which the penalty shall be calculated. The notice shall be transmitted as required by § 66.12. The source owner or operator shall respond to this notice

within 45 days of its receipt and in the form provided in § 66.13.

§ 66.35 Revocation of exemptions.

(a) The Administrator may upon notice and opportunity for a hearing revoke an exemption granted to the owner or operator of any source at any time in accordance with paragraphs (b) and (c) below.

(b) An exemption may be revoked and a penalty for the period of non-exempted noncompliance assessed if:

(1) The grounds for the exemption no longer exist or never did exist, or

(2) In the case of an exemption under § 66.31, the source owner or operator has failed to comply with any interim emission control requirements or schedules of compliance (including increments of progress) contained in the extension, order, suspension or EPA-approved consent decree on which the exemption was based.

(c) The Administrator shall provide the source owner or operator written notice containing the information required by § 66.12 and a statement of the reasons for revocation. The notice shall also specify the date from which the source owner or operator must calculate a penalty. The notice shall be transmitted as required by § 66.12. The source owner or operator shall respond to this notice within 45 days of its receipt and in the form provided in § 66.13.

Subpart E—Decisions on Exemption Requests and Challenges to Notices of Noncompliance

§ 66.41 Decision on petitions.

(a) Within thirty days after receiving a petition filed under § 66.13, the Administrator shall notify the source owner or operator in writing that:

(1) The petition demonstrates that the source owner or operator is entitled to part or all of the relief requested and that the notice of noncompliance is withdrawn or modified accordingly;

(2) The petition does not contain sufficient information to demonstrate that the source owner or operator is entitled to part or all of the relief requested. The Administrator shall

specify what deficiencies exist and request that the source owner or operator supplement his petition within thirty days of receipt of that request. If the petition is not supplemented adequately within this time, or, if supplemented adequately, still fails to demonstrate entitlement to relief, the Administrator shall grant a hearing under paragraph (a)(3) of this section. Any supplemental material provided pursuant to the Administrator's request shall be evaluated as provided in paragraphs (a)(1) and (a)(3) of this section.

(3) A hearing is granted on the issue of whether the source is in violation of applicable legal requirements or is entitled to an exemption under §§ 66.31, 66.32, 66.33, or on both.

[45 FR 50110, July 20, 1980, as amended at 50 FR 36734, Sept. 9, 1985]

§ 66.42 Procedure for hearings.

(a) Except as provided in §§ 66.32 and 66.33, hearings granted under § 66.41(a)(3) shall be held as provided in Subpart J.

(b) If hearings are granted pursuant to both § 66.32 or § 66.33 and under Subpart J, a consolidated hearing in accordance with Subpart J shall be held. At the consolidated hearing the issues that would otherwise have been considered at a hearing under § 66.32 or § 66.33 shall be considered pursuant to the procedures for a hearing provided in those sections.

(c) The Presiding Officer at a hearing granted under § 66.41 shall issue an initial decision within 90 days after the Administrator grants the hearing, unless the duration of the hearing or the deadline for decision is extended by the Presiding Officer upon agreement of the parties. Failure to issue a decision (whether or not by consent) within 90 days shall not affect the validity of the proceedings or the accrual of penalties in any manner.

§ 66.43 Final decision; submission of penalty calculation.

Within forty-five days after EPA has notified the owner or operator of a source of the final Agency decision that it is in violation of applicable legal requirements or is not entitled to an exemption, the owner or operator

shall submit the information required by § 66.13(a), including appropriate compliance and payment schedules and extra interest owed for the period of delay. The penalty shall be calculated from the date of receipt of the original notice of noncompliance.

Subpart F—Review of Penalty Calculation

§ 66.51 Action upon receipt of penalty calculation.

(a) Within thirty days after receipt of a penalty calculation provided pursuant to § 66.13(a)(1) or § 66.43, the Administrator shall notify the source owner or operator in writing, that:

(1) The penalty is provisionally accepted as calculated, subject to any recalculation that may be necessary under § 66.72 after the source has achieved compliance; or

(2) The penalty is incorrect and has been recalculated based on the data provided by the source owner or operator, or other data. The Administrator shall provide a brief statement of the basis for the recalculation and shall identify when and where any supporting data may be examined. The Administrator shall also notify the source owner or operator of the right to petition for a hearing under § 66.52; or

(3) The source owner or operator has not submitted any calculation, or the information submitted is inadequate to enable EPA to verify the owner or operator's penalty calculation. The Administrator shall specify what deficiencies exist and request the source owner or operator to supplement his submission within thirty days of receipt of that request. If an inadequate supplemental submission is made within this time, EPA may calculate the penalty itself or as provided in § 66.22.

(b) Supplemental material provided pursuant to paragraph (a)(3) of this section shall be evaluated as provided in paragraph (a).

§ 66.52 Petitions for reconsideration of calculation.

Within forty-five days after receipt of notice under § 66.51(a)(2) that the

penalty has been recalculated by EPA, a source owner or operator who wishes to challenge EPA's recalculation shall petition in writing for reconsideration. A statement of all arguments on which the owner or operator relies, including all necessary supporting data and a substitute penalty calculation and payment schedule shall be included in or accompany this petition. Issues not raised in the petition shall be deemed waived.

§ 66.53 Decisions on petitions.

Within thirty days after receiving a petition for reconsideration under § 66.52 the Administrator shall:

(a) Accept the penalty calculation of the owner or operator to the extent the Administrator concludes it is correct; or

(b) Grant a hearing to the extent he does not conclude that the petition is correct.

[45 FR 50110, July 20, 1980, as amended at 50 FR 36734, Sept. 9, 1985]

§ 66.54 Procedures for hearing.

(a) Hearings granted under § 66.53 shall be held as provided in Subpart J.

(b) The Presiding Officer at a hearing granted under § 66.53 shall issue an initial decision within ninety days after the Administrator grants the hearing, unless the duration of the hearing or the deadline for decision is extended by the Presiding Officer upon agreement of the parties. Failure to issue a decision (whether or not by consent) within 90 days shall not affect the validity of the proceedings or the accrual of penalties in any manner.

Subpart G—Payment

§ 66.61 Duty to pay.

(a) Except where the owner or operator has submitted a petition pursuant to § 66.13(a)(2), the first installment of the penalty shall be paid on the date six months after receipt of the notice of noncompliance.

(b) Where the source owner has filed a petition pursuant to § 66.13(a)(2), the first installment consisting of payment of penalties for all quarters "missed" as well as for the upcoming

quarter shall be paid on the date six months after a final administrative decision affirming the source owner or operator's liability. Installments shall be paid quarterly thereafter until compliance is achieved. Quarters shall be measured in increments of three calendar months from the date the first payment is due.

(c) A source owner or operator who submits a petition pursuant to § 66.52 shall pay the penalty amount calculated by the owner or operator under § 66.13 or § 66.43 or any penalty calculated by EPA where the owner or operator has failed to calculate such penalty. Within 45 days after EPA has notified the owner or operator of a final administrative action after hearings on such petition, the owner or operator shall submit any necessary modification to the penalty. The revised penalty will be calculated in accordance with the Technical Support Document and the Manual, and a revised schedule, including appropriate adjustments for overpayments or underpayments made, will be established.

§ 66.62 Method of payment.

Payments in excess of \$10,000 under this part shall be made by wire transfer payable to the U.S. Treasury. Payments under this part which are less than \$10,000 shall be made by cashier's or certified check made payable to the United States Treasury, sent by registered mail, return receipt requested, and addressed to the Administrator, Environmental Protection Agency, 401 M Street, S.W., Washington, D.C. 20460. Payment by check is made on the due date if it is postmarked on or before the due date. Payment by wire transfer is made on the due date if the Treasury's account is credited on or before the due date.

§ 66.63 Nonpayment penalty.

(a) Any source owner or operator who fails to make timely payment under § 66.61 shall pay in addition to the penalty owed a quarterly nonpayment penalty. The nonpayment penalty shall be calculated as of the due date of the noncompliance penalty payment and shall be equal to 20 percent of the aggregate amount of the

noncompliance penalties and nonpayment penalties due and owing from the owner or operator on the due date. Partial payments shall be credited first against the nonpayment penalty, then against the noncompliance penalty.

(b) The Administrator shall notify the source owner or operator in writing of the amount of any nonpayment penalty for which the owner or operator is liable. Failure of the Administrator to provide such notice immediately shall not affect the legal obligation of the source owner or operator to pay such penalty. Such nonpayment penalty, as well as the past due noncompliance penalty, shall be payable immediately.

Subpart H—Compliance and Final Adjustment

§ 66.71 Determination of compliance.

(a) An owner or operator of a source who is paying a noncompliance penalty under this part shall notify the Administrator in writing when he believes that the source has come into and is maintaining compliance with all applicable legal requirements. The notice shall be accompanied by any factual data, analytical materials, and legal arguments which the source owner or operator believes support such claim.

(b) Within 30 days of receipt of a source owner's submittal, the Administrator shall determine whether the source has achieved and is maintaining compliance with applicable legal requirements, and shall notify the source owner or operator of this determination in writing. If the Administrator is unable to conclude, on the basis of the information submitted, whether the source has achieved and is maintaining compliance with applicable legal requirements, he shall inform the owner or operator of any additional material that is needed. Within 30 days of receipt of such additional material, the Administrator shall determine whether the source has achieved and is maintaining compliance, and shall notify the source owner or operator of this determination in writing.

(c) If the Administrator determines that the source has not achieved or is

not maintaining compliance with applicable legal requirements, the source owner or operator may petition for reconsideration within 30 days of receipt of the determination. The source owner or operator shall include in this petition any necessary supporting material. Issues not raised in the petition will be deemed waived. The procedures of § 66.41 shall be followed upon the receipt of such petition.

(d) In the event that the applicable legal requirement (as defined in § 66.3(c)) the violation of which forms the basis for the penalty is superseded by another applicable legal requirement (as defined in § 66.3(c)) the owner or operator of a source liable for a noncompliance penalty under this part shall notify the Administrator in writing that the owner or operator believes that the applicable legal requirement is superseded and that the period of noncompliance covered by the notice of noncompliance is ended. The notice shall be accompanied by the legal arguments which the source owner or operator believes support such a claim. Within 30 days of receipt of a source owner or operator's notice, the Administrator shall determine whether the period of covered noncompliance is ended and shall notify the source owner or operator of this determination in writing. In cases where the superseding EPA-approved requirement was not approved by EPA within the time period required by statute, the period of covered noncompliance shall be deemed to have ended on the date when EPA under the statute should have acted.

[45 FR 50110, July 20, 1980 and 50 FR 36734, Sept. 9, 1985]

§ 66.72 Additional payment or reimbursement.

(a) Within 120 days after the source owner or operator receives notification pursuant to § 66.71(b) that it has achieved and is maintaining compliance with applicable legal requirements, or within 120 days after receipt of a decision to that effect upon petition and hearing, or within 120 days after receipt of a decision to that effect upon an appeal to the Administrator, the source owner or operator

shall submit to the Administrator a revised penalty calculation as provided in the Technical Support Document and the Manual, together with data necessary for verification. The revised calculation shall include interest on any underpayment.

(b) Within thirty days after receiving a revised penalty calculation provided pursuant to paragraph (a) of this section, the Administrator shall inform the source owner or operator in writing that:

(1) The revised penalty is correct as calculated;

(2) The revised penalty is incorrect and has been recalculated based on the data provided by the source owner or operator or on other data. The Administrator shall provide to the source owner or operator a brief statement of the basis of the recalculation and shall identify when and where any supporting data may be examined. The Administrator shall also notify the source owner or operator of the right to petition for reconsideration under § 66.73; or

(3) The source owner or operator has not submitted any penalty calculation, or has not submitted enough material to enable EPA to verify the penalty calculation. The Administrator shall specify what deficiencies exist and shall require the source owner or operator to furnish the supplemental material within thirty days of receipt of the notice. The supplemental material submitted will be evaluated in the same manner as the original submittal.

(c) If a source owner or operator fails to submit or to complete a revised penalty calculation when due under this section or the calculation submitted is incorrect, the Administrator may recalculate the penalty or may enter into a contract for independent calculation of the penalty as provided in § 66.22.

(d) Within 120 days after the source owner or operator receives notification pursuant to § 66.71(d) that the period of covered noncompliance ended on the date the applicable legal requirement was superseded (or, in event of EPA delay past an applicable statutory deadline, on the date the applicable legal requirement would have been su-

perseded if there had been no delay past the statutory deadline), the source owner or operator shall submit to the Administrator a revised penalty calculation as provided in the Technical Support Document and Manual together with data necessary for verification. The revised calculation shall include interest on any underpayment. Paragraphs (b) and (c) shall apply to calculations submitted under this paragraph.

[45 FR 50110, July 20, 1980 and 50 FR 36734, Sept. 9, 1985]

§ 66.73 Petition for reconsideration and procedure for hearing.

Within forty-five days of receipt of a notice under § 66.72(b) (2) a source owner or operator may petition for reconsideration in the form and manner provided in § 66.52. The petition shall be evaluated as provided in § 66.53 and any hearing shall be held in conformity with § 66.54.

§ 66.74 Payment or reimbursement.

(a) Within thirty days after any adjustment of a noncompliance penalty under this Subpart has become administratively final:

(1) Any deficiency owed by the source owner or operator shall be paid as provided in § 66.62.

(2) Any reimbursement shall be paid by check from the United States payable to the order of the source owner or operator, and sent by registered or certified mail, return receipt requested.

(b) Any payment under paragraph (a) of this section, shall include interest on the amount of the deficiency or reimbursement due, from the date the deficiency or reimbursement arose, at a rate determined by the Secretary of the Treasury. Such payment shall be calculated in accordance with the Technical Support Document and the Manual.

(c) Any source owner or operator who fails to make timely payment of a deficiency shall pay a nonpayment penalty. The nonpayment penalty shall be calculated as of the due date of the deficiency payment and shall be equal to 20% of the deficiency not paid. Such nonpayment penalty (in ad-

dition to the amount of the deficiency owed) shall be payable immediately. If any part of the nonpayment penalty or deficiency shall remain unpaid at the end of three calendar months from the due date of the deficiency, a further nonpayment penalty shall be due equal to 20% of the sum of all payments due and owing. Partial payments shall be credited first against the nonpayment penalty, then the deficiency.

Subpart I—Final Action

§ 66.81 Final action.

(a) A final Agency action appealable to the courts by the source owner or operator includes and is limited to the following, provided the conditions of paragraph (b) of this section are met:

(1) A notice of determination that a source is in violation of applicable legal requirements;

(2) A notice of decision to deny or revoke an exemption under Subpart D;

(3) A notice of revision by EPA of a penalty calculation or schedule under Subpart F;

(4) A notice of decision by EPA that the source is not in final compliance or any revision by EPA of a final penalty calculation under Subpart H; and

(5) A notice of denial of a petition for reconsideration under § 66.71 or § 66.73.

(6) A decision by the Administrator upon completion of any review of a State action pursuant to Part 67.

(b) The actions listed in paragraph (a) of this section constitute final Agency action only if all administrative remedies have been exhausted. To exhaust administrative remedies a source owner or operator must first petition for reconsideration of the decision in question and, if unsuccessful after hearing or after denial of hearing, appeal the decision in question to the Administrator. The action becomes final upon the completion of review by the Administrator and notice thereof to the owner or operator of the source.

(c) Where a petition seeks reconsideration both of the finding of noncompliance and of the finding of liability on the ground that the source owner

or operator is entitled to an exemption, both questions must be decided before any review by the Administrator is sought, except on agreement of the parties.

[45 FR 50110, July 20, 1980, as amended at 50 FR 36734, Sept. 9, 1985]

Subpart J—Supplemental Rules for Formal Adjudicatory Hearings

§ 66.91 Applicability of supplemental rules.

The Supplemental Rules in this subpart, in conjunction with the Consolidated Rules of Practice (40 CFR Part 22) shall govern all hearings held under this part. To the extent that the provisions of these Supplemental Rules or any other provision of this part are inconsistent with the Consolidated Rules, the provisions of this Part shall govern.

§ 66.92 Commencement of hearings.

(a) The provisions of 40 CFR 22.08 (Consolidated Rules of Practice) shall become applicable when the Administrator grants a hearing.

(b) Upon granting a hearing the Administrator shall immediately transmit to the appropriate Regional Hearing Clerk two copies of the notice granting the hearing and:

(1) In the case of a hearing pursuant to § 66.42, two copies of the notice of noncompliance under § 66.11 (or the revocation notice under § 66.34) and of the petition of the owner or operator under § 66.13, together with supporting documents; and

(2) In the case of a hearing pursuant to § 66.53 or § 66.73, two copies of the penalty calculation of the source owner or operator provided pursuant to § 66.13(a) or § 66.72, and of any Agency recalculation pursuant to § 66.51(a)(2) or § 66.72(b)(2), and of the petition of the source owner or operator for reconsideration pursuant to § 66.52 or § 66.73, together with supporting documents.

(3) The Regional Hearing Clerk shall open and maintain the official file of the proceeding upon receipt of the documents referred to in paragraphs (b)(1) and (2) of this section.

(c) Upon granting a hearing the Administrator shall request the Chief Administrative Law Judge to designate an Administrative Law Judge to serve as the Presiding Officer. The Chief Administrative Law Judge shall make this designation within seven days of receiving the request, and shall notify the Regional Hearing Clerk of his action. The Regional Hearing Clerk shall forward to the Presiding Officer one set of the documents described in paragraph (b).

§ 66.93 Time limits.

The Presiding Officer upon designation shall notify the parties and shall, if appropriate, schedule a prehearing conference (or alternative procedures) under 40 CFR 22.19 and shall notify the parties of the date of hearing under 40 CFR 22.21. The Presiding Officer shall issue an initial decision no later than ninety days after the hearing is granted, unless an extension of the hearing schedule or of the deadline for decision is agreed to by the parties. To that end, the Presiding Officer may establish such deadlines as are reasonable and necessary. Failure to issue a decision within 90 days or further extended deadline (whether or not by consent) shall not affect the validity of the proceedings.

§ 66.94 Presentation of evidence.

(a) In hearings pursuant to § 66.42 EPA shall present evidence of violation of applicable legal requirements. The source owner or operator shall then present any rebuttal evidence.

(b) In hearings under § 66.42 the source owner or operator shall present evidence of entitlement to an exemption. EPA shall then present any rebuttal evidence.

(c) In hearings under §§ 66.54 and 66.73 EPA shall present evidence that its calculation or revisions of the source owner or operator's penalty calculations are correct. The source owner or operator shall then present any rebuttal evidence.

(d) Each matter of controversy shall be determined by the Presiding Officer upon a preponderance of the evidence.

(e) Any documentation submitted pursuant to § 66.92(b) shall automati-

cally be received into evidence in the hearing.

§ 66.95 Decisions of the Presiding Officer: Appeal to the Administrator.

(a) The Presiding Officer shall dispose of the issues raised in the hearing in a single written decision. Such decision shall terminate the Presiding Officer's consideration of those issues.

(b) Penalty calculations and payment schedules, if any, established by the decision of the Presiding Officer shall be based solely on the parameters, terms and conditions of the Technical Support Document, Manual, and Computer Program.

(c) An appeal to the Administrator from a decision of the Presiding Officer shall be made by petition filed within twenty (20) days from receipt by a party of the Presiding Officer's decision. The Administrator shall rule on the appeal within 30 days of receipt of a petition. No appeal may be made before receipt of the decision of the Presiding Officer.

APPENDIX A—TECHNICAL SUPPORT DOCUMENT

NOTE: For text of Appendix A see Appendix A to Part 67.

APPENDIX B—INSTRUCTION MANUAL

NOTE: For text of Appendix B see Appendix B to Part 67.

APPENDIX C—COMPUTER PROGRAM

NOTE: For text of Appendix C see Appendix C to Part 67.

PART 67—EPA APPROVAL OF STATE NONCOMPLIANCE PENALTY PROGRAM

Subpart A—Purpose and Scope

Sec.

67.1 Purpose and scope.

Subpart B—Approval of State Programs

67.11 Standards for approval of State programs.

67.12 Application for approval of programs.

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67.14 Amendments to the program.
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Subpart C—Federal Notice of Noncompliance to Sources in States With Approved Programs

- 67.21 Federal notice of noncompliance to owners or operators of sources in States with approved programs.

Subpart D—EPA Review of State Exemption Decisions

- 67.31 Review by the Administrator.
67.32 Procedure where no formal State hearing was held.
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Subpart E—EPA Review of State Penalty Assessments

- 67.41 When EPA may review.
67.42 Procedure where no formal State hearing was held.
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APPENDIX A—TECHNICAL SUPPORT DOCUMENT
APPENDIX B—INSTRUCTION MANUAL
APPENDIX C—COMPUTER PROGRAM

AUTHORITY: Sec. 120 of the Clean Air Act, as amended, 42 U.S.C. 7420.

SOURCE: 45 FR 50117, July 28, 1980, unless otherwise noted.

Subpart A—Purpose and Scope

§ 67.1 Purpose and scope.

This part describes the standards and procedures under which EPA will approve State programs for administering the noncompliance penalty program under section 120 of the Clean Air Act and will evaluate actions taken by States with approved programs. Subpart A describes the purpose of the part. Subpart B states the conditions under which EPA will approve State programs to administer the noncompliance penalty provisions. Subparts C and D state when and how EPA will issue its own notices to owners or operators of sources in States with approved programs, and how it will review State decisions to grant or deny exemptions from the penalty. Finally, Subpart E states how EPA will review State assessments of a penalty.

Subpart B—Approval of State Programs

§ 67.11 Standards for approval of State programs.

(a) The Administrator shall approve any program submitted by a State, or by a local governmental agency where no program has been submitted by a State, for administering the noncompliance penalty provisions of section 120 of the Clean Air Act upon finding that the program conforms to the requirements of the Act and to those of this part and 40 CFR Part 66. References to "State program" in this part shall be read as including local governmental agencies and their programs.

(b) The Administrator shall not approve any State program that does not provide explicitly for:

(1) Issuance of a notice of noncompliance, in a manner consistent with procedures under Part 66, upon discovery by the State or upon notification by EPA of a violation of applicable legal requirements, which notice satisfies the informational requirements set forth in § 66.13.

(2) Levels of staffing and funding satisfactory, in the judgment of the Administrator, to implement and enforce the requirements of section 120 in that State, together with adequate provision for maintaining such levels;

(3) A capability to carry out the financial analysis and procedures specified in these regulations and the Technical Support Document, Instruction Manual, and related Computer Program, available from the Director of Stationary Source Compliance Division, EN-341, 401 M Street, NW., Washington, DC 20460, together with adequate provision for maintaining such capability. Such capability may be provided by trained State personnel or through qualified contractors;

(4) Except as provided in paragraph (a)(6) of this section, an administrative hearing whenever the owner or operator of a source submits a petition for reconsideration of a notice of noncompliance on the ground that the source either is not in violation of applicable legal requirements, or is entitled to an exemption, or both, or submits a petition to challenge a recalculation of the

penalty by the State, provided that such petitions raise issues of fact that would require a hearing under Part 66. This hearing need not conform to the requirements of 5 U.S.C. 554 as long as its procedures provide for:

(i) An initial decision by the hearing officer on the record;

(ii) A hearing officer who has not performed investigative or litigating functions in any enforcement action against the source owner or operator in question;

(iii) Opportunity for public participation on reasonable notice, including intervention, by interested persons;

(iv) Opportunity for cross-examination or an equivalent opportunity for confrontation between persons advocating differing positions on material factual matters; and

(v) An initial decision by the hearing officer within ninety days of commencement of the hearing unless such period is extended upon agreement of the parties.

(5) Explicit provision for:

(i) notice to the Administrator of any determination granting an exemption, or finding a source in violation of applicable legal requirements, and any penalty calculation and payment schedule approved or calculated by the State, together with any information necessary to verify its accuracy;

(ii) within 30 days of receipt of a request from the Administrator, transmission of a copy of the record of the hearing held under paragraphs (a)(4) or (6) of this section, including any proffered evidence and a ruling on its admissibility and the State's decision on the merits; and

(iii) additional reporting and record keeping, if necessary, adequate to enable the Administrator to review the State's administration of the program and determine whether it conforms to the Act and to Part 66 of these regulations. Such requirements will be specified in the Notice of Delegation to the State.

(6) A hearing on the question of whether the owner or operator of a source is entitled to an exemption pursuant to §§ 66.32 or 66.33 may be informal. The hearing shall be scheduled upon notice to the public. Reasonable opportunity to testify and for submission

of questions to the petitioner by members of the public shall be afforded. A record of the hearing shall be made, and the decision of the hearing officer made in writing within a reasonable period of time after the close of the hearing.

(c) The State may delegate all or part of its responsibilities under its program to a local governmental agency to implement the program within the jurisdiction of the local agency, *Provided* that the program of the local government agency meets the requirements of this section.

(d) No State penalty program or program of one of its agents shall be disapproved because it is more stringent than the program established by Part 66 or by Section 120 where the State or local agent concludes that it has independent authority under State or local law to implement and administer the more stringent portions of the program.

[45 FR 50117, July 28, 1980, as amended at 54 FR 25259, June 14, 1989]

§ 67.12 Application for approval of program.

A state that wishes to administer a Section 120 program shall submit an application in writing to the Administrator describing its proposed program. All necessary supporting materials shall accompany the application.

§ 67.13 Approval.

(a) The Administrator shall evaluate any application submitted under § 67.12 and shall:

(1) Approve the program and delegate authority to the State to administer the program if he determines that the requirements of § 67.11 have been and will be met; or

(2) Request additional information if he determines that the information submitted is not sufficient to allow him to determine whether the requirements of § 67.11 have been and will be met; or

(3) Disapprove the State program if he determines that the information submitted establishes that the requirements of § 67.11 have not been or will not be met.

(b) The Administrator shall notify the State in writing of his action under paragraph (a) of this section and shall state the reasons for his action.

(c) In all cases of delegation (whether or not express provision is made in the notice of delegation) the Administrator shall retain continuing authority to issue notices of noncompliance, review exemption requests or penalty calculations, or take any other steps set forth in Part 66 to assess and collect these penalties. Such authority shall be exercised pursuant to the provisions of § 67.21.

(d) The Administrator shall retain exclusive authority to assess and collect penalties against source owners or operators of facilities in the State who were issued notices of noncompliance pursuant to Part 66 prior to the effective date of the delegation, except to the extent the Administrator specifically delegates such authority to the State.

§ 67.14 Amendments to the program.

A State or local agent with a program approved pursuant to § 67.13 may propose amendments to that program to the Administrator. The Administrator shall evaluate whether the State or local agent's program as amended would conform to the requirements of § 67.11 and shall respond as provided in § 67.13.

§ 67.15 Revocation.

If the Administrator determines that a State with a program approved under § 67.13 is not administering the program in conformity with the requirements of the Act or § 67.11, or the delegation of authority, he shall provide the State written notice of that determination, setting forth his reasons. Copies of all supporting materials shall accompany the notice if requested, or shall be placed on file in the appropriate Regional Office and made available for inspection during normal business hours. The State shall have 90 days in which to respond in writing to this determination. If the Administrator finds after reviewing the State response that (a) the State is in fact administering the program in conformity with § 67.11, or (b) there are reasonable grounds to believe the

State program will immediately be brought into conformity with that section, he shall withdraw his determination. If he finds that neither of these conditions has been met, he shall withdraw the delegation of authority to the State.

Subpart C—Federal Notice of Noncompliance to Sources in States With Approved Programs

§ 67.21 Federal notice of noncompliance to owners or operators of sources in States with approved programs.

(a) The Administrator shall issue a notice of noncompliance to the owner or operator of any source in a State with an approved program if he determines that the State or its local agent has failed to issue such notice, provided that he shall first give 30 days notice to the State of his intent to issue a notice of noncompliance to the owner or operator of the source in question unless the State or its agent does so first. Any notice issued by the Administrator pursuant to this section shall be deemed to be issued pursuant to the provisions of Part 66.

(b) The issuance of a notice of noncompliance shall operate to withdraw EPA delegation of authority to the State with respect to the particular facility in question.

(c) If the Administrator determines that the State or local agent has issued a notice of noncompliance but has failed to pursue diligently subsequent steps for the assessment and collection of the penalty, he shall notify the State of his intent to withdraw delegation of authority to the State with respect to the facility in question and take appropriate actions pursuant to Part 66 unless the State or local agent, within 30 days, takes appropriate action in accordance with the requirements of this part. In either case the penalty will be calculated from the date of the State notice.

Subpart D—EPA Review of State Compliance or Exemption Decisions

§ 67.31 Review by the Administrator.

(a) The Administrator may, on his own initiative, review any determination by a State or its agent that a source owner or operator is or is not in compliance with applicable legal requirements or is or is not entitled to an exemption, to determine whether that determination conforms to the requirements of the Act and Part 66 (as modified by § 67.11).

(b) The Administrator shall review any such determination upon receipt of a petition alleging that the State's determination does not conform to the requirements of the Act and Part 66 (as modified by § 67.11). Such petition must be filed within 20 days of issuance of the State's decision.

(c) The Administrator shall give notice in writing to the State or local agent, to the owner or operator of the source, and to the petitioner of his intent to review the determination. Such notice shall be given within 90 days of the Administrator's receipt of the State or local agent's determination. Unless otherwise provided, such notice shall not withdraw EPA's delegation of authority to the State or local agent over the particular facility in question.

(d) No such State determination shall become final until the expiration of 90 days after the Administrator's receipt of the notice required by § 67.11(b)(5).

(1) If the Administrator does not issue a notice of intent to review within that period, the State determination shall, upon expiration of such period, constitute final action of the Administrator under section 120 of the Act.

(2) If the Administrator issues a notice of intent to review within that period, the State determination shall not become final until the Administrator takes final action after reviewing the determination.

(e) Except as otherwise provided, a State determination shall be approved if there was a reasonable basis in law and in fact for making the determination.

§ 67.32 Procedure where no formal State hearing was held.

(a) In reviewing a decision that a source is in compliance with applicable legal requirements or entitled to an exemption for which no hearing conforming to § 67.11(b) (4) or (6) was held, the Administrator shall evaluate the accuracy and adequacy of the documents transmitted to him pursuant to § 67.11(b)(5) and shall invite submission of comments on issues identified by him as relevant to his review.

(b) If the Administrator concludes that no hearing need have been held and that the State determination was correct, he shall notify the State, the source owner or operator, and other participants of his determination, which shall constitute final agency action by EPA under authority of section 120. If the Administrator concludes that the petition of the source owner or operator presented information which, if true, would have altered the owner or operator's liability for a penalty, he shall upon notice to the State or local agent schedule a hearing in accordance with Subpart E of Part 66. Such notice shall operate as a withdrawal of EPA's delegation of authority to the State or local agent over the facility in question unless the State or local agent schedules a hearing within 15 days of receipt of the notice.

(c) If the Administrator concludes that the State determination did not conform to the requirements of the Act or of Part 66 (as modified by § 67.11), he shall by written notice revoke the determination. Such revocation shall operate as a withdrawal of EPA's delegation of authority to the State or local agent over the facility in question. The source owner or operator may then petition for review of the Administrator's decision pursuant to the provisions of § 66.13.

(d) Unless otherwise provided in the Administrator's notice to the State or local agent, any noncompliance penalties owed by the source owner or operator shall be paid to the State or local agent. The Administrator shall send a copy of this notice to the source owner or operator.

§ 67.33 Procedure where a formal State hearing was held.

(a) In reviewing a decision that a source is in compliance with applicable legal requirements or is entitled to an exemption for which a hearing conforming to § 67.11(b) (4) or (6) was held, the Administrator may invite comment on issues identified by him as relevant to his review and shall propose or make findings as to the correctness of the determination and the accuracy and adequacy of the material transmitted pursuant to § 67.11(b)(5).

(b) The Administrator shall notify all participants in the State hearing of his findings and conclusions. If the Administrator concludes that the State determination conformed to the requirements of the Act and of Part 66 (as modified by § 66.11), the Administrator's determination shall constitute final administrative action by EPA under authority of Section 120. If the Administrator finds that the State determination did not conform to the requirements of the Act and of Part 66 (as modified by § 67.11), the findings shall constitute proposed findings and the notice shall invite participants to file exceptions thereto. If the Administrator considers it desirable, he may schedule a time for argument.

(c) Within 60 days of receipt of any briefs or exceptions or after oral argument pursuant to paragraph (b), the Administrator shall affirm, modify, or revoke his proposed findings that the State's determination did not conform to the requirements of the Act or of Part 66 (as modified by § 67.11). The decision shall be in writing. Notice and a copy of the decision shall be provided to the source owner or operator and to all other participants in the State hearing. The decision shall constitute a final administrative action by EPA under authority of section 120.

(d) If the Administrator finds that deficiencies in the hearing record prevent him from determining whether the determination of the State or local agent conformed to the requirements of the Act and Part 66 (as modified by § 67.11), he shall notify the State or local agent of his finding and specify what deficiencies exist and schedule a hearing pursuant to Subpart E of Part 66. Such notice shall operate as a

withdrawal of EPA's delegation of authority to the State or local agent over the facility in question unless the State or local agent schedules a supplemental hearing to correct the deficiencies within 15 days of receipt of the notice.

(e) If the Administrator concludes that the source is in violation of applicable legal requirements or is not entitled to an exemption, or both, and unless otherwise ordered in the decision, the source owner or operator shall submit a penalty calculation to the State within 45 days of receipt of the notice of determination.

Subpart E—EPA Review of State Penalty Assessments

§ 67.41 When EPA may review.

(a) The Administrator may on his own initiative or on petition review any initial, interim, or final penalty calculation made or approved by the State or local agent to determine whether it conforms to the requirements of the Act, of Part 66, of the Technical Support Document and the Instruction Manual. The Administrator shall notify the State or local agent in writing of his intention to review the calculation within 60 days of receipt by EPA of the calculation or any item considered by the State in making or approving such calculation, whichever occurs later.

(b) No such State¹ determination shall become final until the expiration of 90 days after the Administrator's receipt of the notice required by § 67.11(b)(5).

(1) If the Administrator does not issue a notice of intent to review within that period, the State determination shall, upon expiration of such period, constitute final action of the Administrator under Section 120 of the Act.

(2) If the Administrator issues a notice of intent to review within that period, the State determination shall not become final until the Administrator takes final action after reviewing the determination.

(c) Except as otherwise provided, a State determination shall be approved if there was a reasonable basis in law

and in fact for making the determination.

§ 67.42 Procedure where no formal State hearing was held.

(a) In reviewing a penalty calculation for which no hearing conforming to the requirements of § 67.11(b)(4) was held, the Administrator shall evaluate the accuracy and adequacy of the data contained in the documents transmitted to him pursuant to § 67.11(b)(5) and shall invite comments on issues identified by him as relevant to his review.

(b) If the Administrator concludes that no hearing need have been held and that the State determination was correct, he shall notify the State, the source owner or operator, and other participants of his determination, which shall constitute final agency action by EPA under authority of Section 120. If the Administrator concludes that the petition of the source owner or operator for reconsideration of a recalculation presented information which, if true, would have altered the amount of the penalty calculated, he shall upon notice to the State schedule a hearing in accordance with Subpart F of Part 66. Such notice shall operate as a withdrawal of EPA's delegation of authority to the State or local agent over the facility in question unless the State or local agent schedules a hearing within 15 days of receipt of the notice.

(c) If the Administrator concludes that the determination of the State or local agent not to hold a hearing was proper but that the penalty calculation does not conform to the requirements of the Act or of Part 66, he shall by written notice revoke the determination and issue a notice of recalculation to the source owner or operator pursuant to § 66.51. A copy of the notice of recalculation shall be provided to the State or local agent. The notice of recalculation shall constitute final administrative action by EPA under authority of Section 120 unless the source owner or operator petitions for reconsideration under § 66.52, in which case it shall operate as a withdrawal by EPA of its delegation of authority to the State or local agent over the facility in question.

(d) Unless otherwise provided in the Administrator's notice, noncompliance penalties finally determined to be owed shall be paid to the State or local agent.

§ 67.43 Procedure where a formal State hearing was held.

(a) In reviewing a penalty calculation for which a hearing conforming to § 67.11(b)(4) was held, the Administrator may invite comment on issues identified by him as relevant to his review and shall propose or make findings as to the correctness of the determination and shall evaluate the accuracy and adequacy of the material transmitted pursuant to § 67.11(b)(5).

(b) The Administrator shall notify all participants in the State hearing of his findings and conclusions. If the Administrator finds that the State determination conformed to the requirements of the Act, Part 66 (as modified by § 67.11), the Technical Support Document, and the Instruction Manual, his determination shall constitute a final action pursuant to Section 120. If the Administrator finds that the State determination did not conform to the requirements of the Act or of Part 66 (as modified by § 67.11) or to the Technical Support Document or Instruction Manual, the findings shall constitute proposed findings, and the notice shall invite participants to file exceptions to his proposed findings and, if necessary, schedule a time for argument.

(c) Within 60 days of receipt of any briefs or exceptions or after oral argument, the Administrator shall affirm, modify, or revoke his proposed findings that the State or local agent's determination did not conform to the requirements of the Act or of Part 66 (as modified by § 67.11) or the Technical Support Document or Instruction Manual. The decision shall be in writing. Notice and a copy of the decision, which shall constitute final administrative action by EPA pursuant to Section 120, shall be provided to the source owner or operator and to all other participants in the State hearing.

(d) If the Administrator finds that deficiencies in the State or local

agent's hearing record prevent him from determining whether the State or local agent's determination conformed to the requirements of the Act and Part 66 (as modified by § 67.11) or the Technical Support Document or Instruction Manual, he shall notify the State or local agent of his decision and specify what deficiencies exist and schedule a hearing in accordance with Subpart F of Part 66. Such notice shall operate to withdraw EPA's delegation of authority to the State or local agent over the facility in question unless the State or local agent within 15 days schedules a supplemental hearing to correct the deficiencies.

(e) Unless otherwise provided in the Administrator's notice to the State or local agent, any noncompliance penalties owed by the source owner or operator shall be paid to the State or local agent.

APPENDIX A—TECHNICAL SUPPORT DOCUMENT

NOTE: EPA will make copies of Appendix A available from: Director, Stationary Source Compliance Division, EN-341, 401 M Street, SW., Washington, DC 20460.

[54 FR 25259, June 20, 1989]

APPENDIX B—INSTRUCTION MANUAL

NOTE: EPA will make copies of Appendix B available from: Director, Stationary Source Compliance Division, EN-341, 401 M Street, SW., Washington, DC 20460.

[54 FR 25259, June 20, 1989]

APPENDIX C—COMPUTER PROGRAM

NOTE: EPA will make copies of Appendix C available from: Director, Stationary Source Compliance Division, EN-341, 401 M Street, SW., Washington, DC 20460.

[54 FR 25259, June 20, 1989]

PART 69—SPECIAL EXEMPTIONS FROM REQUIREMENTS OF THE CLEAN AIR ACT

Subpart A—Guam

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69.11 New exemptions. [Reserved]
69.12 Continuing exemptions.

Sec.

Subpart B—American Samoa [Reserved]

69.21 New exemptions. [Reserved]

Subpart C—Commonwealth of the Northern Mariana Islands [Reserved]

69.31 New exemptions. [Reserved]

AUTHORITY: Sec. 325(b), Clean Air Act, as amended (42 U.S.C. 7625-1)

SOURCE: 50 FR 25577, June 20, 1985, unless otherwise noted.

Subpart A—Guam

§ 69.11 New exemptions. [Reserved]

§ 69.12 Continuing exemptions.

(a) Effective on the expiration date of the initial eighteen month exemption provided under section 325(b) of "the Act", the Administrator of the Environmental Protection Agency (EPA) exempts the Guam Power Authority's two sixty-six megawatt oil-fired steam units which comprise the Cabras Power Plant from sulfur dioxide requirements associated with New Source Performance Standards (NSPS) under section 111 of the Clean Air Act and from the related NSPS limitation on sulfur dioxide emissions contained in the Guam SIP.

(b) The exemption will be reviewed at intervals and upon occasions to be specified by EPA (not longer than 2 years), allowing EPA to determine whether the factual circumstances upon which it is based, including commitments made by GPA in the application for extension and the continuing attainment of the National Ambient Air Quality Standards (NAAQS) for Sulfur Dioxide, have changed. The commitments include reporting requirements specified by the Guam Environmental Protection Agency (GEPA), including but not limited to strict implementation of both the monitoring (wind direction and ambient SO₂ concentration) and fuel switching portions of the control strategy, reporting to GEPA of all applications of the strategy, and reporting to GEPA of laboratory analyses of percent sulfur in all new fuel stocks acquired GPA. A finding by EPA that the source is not in compliance with the terms of the exemption will be

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grounds for enforcement of the terms of the exemption under section 113. A finding by EPA that factual circumstances have changed will be grounds for revocation of the exemption and enforcement of the underlying Clean Air Act requirements.

(c) It is a condition of this action that GPA provide to EPA a copy of any GPA application for rate changes or for commercial credit for construction or replacement of capital assets, simultaneously with submission of such application to the rate making authority or commercial credit institution. No later than the 90th day after a finding by EPA that the circumstances upon which the determination for continuing the exemption was originally made have changed, this exemption shall terminate unless within that time GPA submits information that it is taking all practicable steps to comply with NSPS and SIP requirements related to SO₂. EPA shall review such information under the procedures it has established and shall, as appropriate, extend or terminate the exemption.

Subpart B—American Samoa [Reserved]

§ 69.21 New exemptions. [Reserved]

Subpart C—Commonwealth of the Northern Mariana Islands [Re- served]

§ 69.31 New exemptions. [Reserved]

PART 79—REGISTRATION OF FUELS AND FUEL ADDITIVES

Subpart A—General Provisions

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- 79.2 Definitions.
- 79.3 Availability of information.
- 79.4 Requirement of registration.
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Subpart B—Fuel Registration Procedures

- 79.10 Notification of fuel manufacturer.
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- 79.13 Registration.
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Subpart C—Additive Registration Procedures

- 79.20 Notification by additive manufacturer.
- 79.21 Information and assurances to be provided by the additive manufacturer.
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Subpart D—Designation of Fuels and Additives

- 79.30 Scope.
- 79.31 Additives.
- 79.32 Motor vehicle gasoline.
- 79.33 Motor vehicle diesel fuel.

AUTHORITY: Secs. 211 and 301(a) of the Clean Air Act, 42 U.S.C. 1857f-6c and 1857g.

SOURCE: 40 FR 52011, Nov. 7, 1975, unless otherwise noted.

Subpart A—General Provisions

§ 79.1 Applicability.

The regulations of this part apply to the registration of fuels and fuel additives designated by the Administrator pursuant to section 211 of the Clean Air Act (42 U.S.C. 1857f-6c, as amended by section 9, Pub. L. 91-604).

§ 79.2 Definitions.

As used in this part, all terms not defined herein shall have the meaning given them in the Act:

(a) "Act" means the Clean Air Act (42 U.S.C. 1857 et seq., as amended by Pub. L. 91-604).

(b) "Administrator" means the Administrator of the Environmental Protection Agency.

(c) "Fuel" means any material which is capable of releasing energy or power by combustion or other chemical or physical reaction.

(d) "Fuel manufacturer" means any person who, for sale or introduction into commerce, produces or manufactures a fuel or causes or directs the alteration of the chemical composition

of, or the mixture of chemical compounds in, a bulk fuel by adding to it an additive.

(e) "Additive" means any substance, other than one composed solely of carbon and/or hydrogen, that is intentionally added to a fuel named in the designation (including any added to a motor vehicle's fuel system) and that is not intentionally removed prior to sale or use.

(f) "Additive manufacturer" means any person who produces or manufactures an additive for use as an additive and/or sells an additive under his own name.

(g) "Range of concentration" means the highest concentration, the lowest concentration, and the average concentration of an additive in a fuel.

(h) "Chemical composition" means the name and percentage by weight of each compound in an additive and the name and percentage by weight of each element in an additive.

(i) "Chemical structure" means the molecular structure of a compound in an additive.

(j) "Impurity" means any chemical element present in an additive that is not included in the chemical formula or identified in the breakdown by element in the chemical composition of such additive.

§ 79.3 Availability of information.

The availability to the public of information provided to, or otherwise obtained by, the Administrator under this part shall be governed by Part 2 of this chapter.

[41 FR 36918, Sept. 1, 1976]

§ 79.4 Requirement of registration.

(a) *Fuels.* (1) No manufacturer of any fuel designated under this part shall, after the date prescribed for such fuel in this part, sell, offer for sale, or introduce into commerce such fuel unless the Administrator has registered such fuel.

(2) No manufacturer of a registered fuel shall add or direct the addition to it of an additive which he has not previously reported unless he has notified the Administrator of such intended use, including the expected or estimated range of concentration. If necessary to meet an unforeseen production

problem, however, a fuel manufacturer may use an additive that he has not previously reported provided that (i) the additive is on the current list of registered additives and (ii) the fuel manufacturer notifies the Administrator within 30 days regarding such unforeseen use and his plans regarding continued use, including the expected or estimated range of concentration.

(3) Any designated fuel that is (i) in a research, development, or test status; (ii) sold to automobile, engine, or component manufacturers for research, development, or test purposes; or (iii) sold to automobile manufacturers for factory fill, and is not in any case offered for commercial sale to the public, shall be exempt from registration.

(4) A domestic fuel manufacturer may purchase and offer for commercial sale foreign-produced fuel containing unidentified additives provided that within 30 days of his offer for sale he notifies the Administrator of the purchase, the source of purchase, the quantity purchased, and summarized results of any tests performed to determine the acceptability of the purchased fuel to the fuel manufacturer.

(b) *Additives.* (1) No manufacturer of any additive designated under this part shall, after the date prescribed for such additive in this part, sell, offer for sale, or introduce into commerce such additive for use as an additive unless the Administrator has registered such additive.

(2) Any designated additive that is either (i) in a research, development, or test status or (ii) sold to petroleum, automobile, engine, or component manufacturers for research, development, or test purposes, and in either case is not offered for commercial sale to the public, shall be exempt from registration.

(3) Process chemicals used by refineries during the refinery process are exempted from the requirement for registration.

(4) If an additive manufacturer prepares for sale only to fuel manufacturers (i) a blend or mixture of two or more registered additives or (ii) a blend or mixture of one or more registered additives with one or more substances containing only carbon and/or

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hydrogen, he will not be required to register such blend or mixture provided he will, upon request, furnish the Administrator with the names and percentages by weight of all components of such blend or mixture.

[40 FR 52011, Nov. 7, 1975, as amended at 41 FR 21324, May 25, 1976]

§ 79.5 Periodic reporting requirements.

(a) *Fuel manufacturers.* (1) For each calendar quarter (January through March, April through June, July through September, October through December) commencing after the date prescribed for a particular fuel in Subpart D, fuel manufacturers shall submit to the Administrator a report for each registered fuel showing (i) the range of concentration of each additive reported under § 79.11(a) and (ii) the volume of such fuel produced in the quarter. Reports shall be submitted within 45 days after the close of the reporting period on forms supplied by the Administrator upon request.

(2) Fuel manufacturers shall submit to the Administrator a report annually for each registered fuel providing additional data and information as specified in § 79.31(c) and (d) in the designation of the fuel in Subpart D. Reports shall be submitted on or before March 31 for the preceding year or part thereof on forms supplied by the Administrator upon request. If the date prescribed for a particular fuel in Subpart D or the later registration of a fuel is between October 1 and December 31, no report will be required for the period to the end of that year.

(b) *Additive manufacturers.* Additive manufacturers shall submit to the Administrator a report annually for each registered additive providing additional data and information as specified in paragraphs (c) and (d) in the designation of the additive in Subpart D. Additive manufacturers shall also report annually the volume of each additive produced. Reports shall be submitted on or before March 31 for the preceding year or part thereof on forms supplied by the Administrator upon request. If the date prescribed for a particular additive in Subpart D or the later registration of an additive is between October 1 and December 31, no

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report will be required for the period to the end of that year. These periodic reports shall not, however, be required for any additive that is:

(1) An additive registered under another name,

(2) A blend or mixture of two or more registered additives, or

(3) A blend or mixture of one or more registered additives with one or more substances containing only carbon and/or hydrogen.

§ 79.6 Requirement for testing.

(a) The Administrator may establish procedures and protocols for the conduct of tests to determine potential public health effects of a designated fuel or additive (including, but not limited to, carcinogenic, teratogenic, or mutagenic effects) and may thereafter require a fuel manufacturer or an additive manufacturer to conduct tests in conformance with such test procedures and protocols.

(b) In order to obtain such information as is reasonable and necessary to determine the emissions resulting from the use of a fuel or an additive in a fuel, the effect of such fuel or additive on the emission control performance of any motor vehicle or motor vehicle engine, or the extent to which such emissions affect the public welfare, the Administrator may require a fuel manufacturer or an additive manufacturer to conduct tests in accordance with protocols based on generally accepted test methods and procedures.

(c) Any test required under this section shall be performed in accordance with a time schedule prescribed by the Administrator, after consultation with the manufacturer involved.

(d) The Administrator may require a fuel manufacturer to perform tests under this section on any fuel registered for such manufacturer or an additive manufacturer to perform tests under this section on any additive registered for such manufacturer. He may not require any test as a prerequisite of registration unless it is part of a test protocol established by regulation a reasonable time prior to notification by the manufacturer.

§ 79.7 Samples for test purposes.

When the Administrator requires for test purposes a fuel or additive which is not readily available in the open market, he may request the manufacturer of such fuel or additive to furnish a sample in a reasonable quantity. The fuel or additive manufacturer shall comply with such request within 30 days.

§ 79.8 Penalties.

Any person who violates section 211(a) of the Act or who fails to furnish any information required under this part shall forfeit and pay to the United States a civil penalty of \$10,000 for each and every day of the continuance of such violation, which shall accrue to the United States and be recovered in a civil suit in the name of the United States, brought in the district in which such person does business. The Administrator may, upon application therefor, remit or mitigate any such forfeiture; and he shall have authority to determine the facts upon all such applications.

Subpart B—Fuel Registration Procedures

§ 79.10 Notification of fuel manufacturer.

Any manufacturer of a designated fuel who wishes to have such fuel registered shall notify the Administrator in accordance with § 79.11 at least 60 days prior to the date prescribed for such fuel in Subpart D or, after such prescribed date, at least 30 days prior to the date on which such fuel manufacturer proposes to begin to sell, offer for sale, or introduce into commerce such fuel. If a fuel manufacturer produces more than one grade or brand of a designated fuel, only one notification or report is required for that designated fuel, showing highest, lowest, and average values for all such grades or brands. Each notification shall be signed by the fuel manufacturer or his agent and shall be submitted on such forms as the Administrator will supply on request.

§ 79.11 Information and assurances to be provided by the fuel manufacturer.

Each notification submitted by a fuel manufacturer shall include the following:

(a) The commercial identifying name of each additive that will or may be used in a designated fuel subsequent to the date prescribed for such fuel in Subpart D;

(b) The name of the additive manufacturer of each additive named;

(c) The range of concentration of each additive named, as follows:

(1) In the case of an additive which has been or is being used in the designated fuel, the range during any 3-month or longer period prior to the date of submission;

(2) In the case of an additive which has not been used in the designated fuel, the expected or estimated range;

(d) The purpose-in-use of each additive named;

(e) The description (or identification, in the case of a generally accepted method) of a suitable analytical technique (if one is known) that can be used to detect the presence of each named additive in the designated fuel and/or to measure its concentration therein;

(f) Such other data and information as are specified in the designation of the fuel in Subpart D;

(g) Assurances that the fuel manufacturer will notify the Administrator in writing and within a reasonable time of any change in:

(1) The name of any additive previously reported;

(2) The name of the manufacturer of any additive being used;

(3) The purpose-in-use of any additive;

(4) Information submitted pursuant to paragraph (e) of this section;

(h) Assurances that the fuel manufacturer will not represent, directly or indirectly, in any notice, circular, letter, or other written communication, or any written, oral, or pictorial notice or other announcement in any publication or by radio or television, that registration of the fuel constitutes endorsement, certification, or approval by any agency of the United States.

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§ 79.12 Determination of noncompliance.

Whenever the Administrator determines that a notification fails to comply with the regulations of this part, he shall within 30 days of receipt of the notification (in the case of registration prior to the date prescribed for the fuel in Subpart D, by such prescribed date) inform the noncomplying fuel manufacturer of the reasons for such determination.

[41 FR 21324, May 25, 1976]

§ 79.13 Registration.

(a) If the provisions of this part requiring the submission of information and the giving of assurances have been complied with for a particular fuel, the Administrator shall, within 30 days of receipt of the notification (in the case of registration prior to the date prescribed for the fuel in Subpart D, by such prescribed date), register that fuel and notify the fuel manufacturer of such registration.

(b) The Administrator shall maintain a list of registered fuels, which shall be available to the public upon request.

[40 FR 52011, Nov. 7, 1975, as amended at 41 FR 21324, May 25, 1976]

§ 79.14 Termination of registration of fuels.

Registration may be terminated by the Administrator if the fuel manufacturer requests such termination in writing.

Subpart C—Additive Registration Procedures

§ 79.20 Notification by additive manufacturer.

Except as provided in § 79.23(b), any manufacturer of a designated additive who wishes to have such additive registered shall notify the Administrator in accordance with § 79.21 at least 150 days prior to the date prescribed for such additive in Subpart D or, after such prescribed date, at least 30 days prior to the date on which such additive manufacturer proposes to begin to sell, offer for sale, or introduce into commerce such additive. Each notification shall be signed by the additive

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manufacturer or his agent and shall be submitted on such forms as the Administrator will supply upon request.

[40 FR 52011, Nov. 7, 1975, as amended at 41 FR 21324, May 25, 1976]

§ 79.21 Information and assurances to be provided by the additive manufacturer.

Each notification submitted by an additive manufacturer shall include the following:

(a) The chemical composition of the additive with the methods of analysis identified, except that

(1) If the chemical composition is not known, full disclosure of the chemical process of manufacture will be accepted in lieu thereof;

(2) In the case of an additive for engine oil, only the name, percentage by weight, and method of analysis of each element in the additive are required provided, however, that a percentage figure combining the percentages of carbon, hydrogen, and/or oxygen may be provided unless the breakdown into percentages for these individual elements is already known to the registrant.

(3) In the case of a purchased component, only the name, manufacturer, and percent by weight of such purchased component are required if the manufacturer of the component will, upon request, furnish the Administrator with the chemical composition thereof.

(b) The chemical structure of each compound in the additive if such structure is known and is not adequately specified by the name given under "chemical composition." Nominal identification is adequate if mixed isomers are present.

(c) The description (or identification, in the case of a generally accepted method) of a suitable analytical technique (if one is known) that can be used to detect the presence of the additive in any fuel named in the designation and/or to measure its concentration therein.

(d) Fuels in which the use of the additive is recommended and the manufacturer's recommended range of concentration and purpose-in-use in each.

(e) Such other data and information as are specified in the designation of the additive in Subpart D.

(f) Assurances that any change in information submitted pursuant to (1) paragraphs (a), (b), (c), and (d) of this section will be provided to the Administrator in writing within 30 days of such change; and (2) paragraph (e) of this section as provided in § 79.5(b).

(g) Assurances that the additive manufacturer will not represent, directly or indirectly, in any notice, circular, letter, or other written communication or any written, oral, or pictorial notice or other announcement in any publication or by radio or television, that registration of the additive constitutes endorsement, certification, or approval by any agency of the United States.

[40 FR 52011, Nov. 7, 1975, as amended at 41 FR 21324, May 25, 1976]

§ 79.22 Determination of noncompliance.

Whenever the Administrator determines that a notification fails to comply with the regulations of this part, he shall within 30 days of the receipt of the notification (in the case of registration prior to the date prescribed for the additive in Subpart D, by such prescribed date) inform the noncomplying additive manufacturer of the reasons for such determination.

[41 FR 21324, May 25, 1976]

§ 79.23 Registration.

(a) If the provisions of this part requiring the submission of information and the giving of assurances have been complied with for a particular additive, the Administrator shall, within 30 days of receipt of the notification (in the case of registration prior to the date prescribed for the additive in Subpart D, by such prescribed date), register that additive and notify the additive manufacturer of such registration.

(b) Any additive which had been registered under the provisions of this part prior to the promulgation date of these regulations shall be deemed registered under these regulations upon such promulgation date. The information requirements of § 79.21 must be complied with for any such additive

within 6 months after the promulgation of this part.

(c) The Administrator shall maintain a list of registered additives, which shall be available to the public upon request.

[40 FR 52011, Nov. 7, 1975, as amended at 41 FR 21324, May 25, 1976]

§ 79.24 Termination of registration of additives.

Registration may be terminated by the Administrator if the additive manufacturer requests such termination in writing.

Subpart D—Designation of Fuels and Additives

§ 79.30 Scope.

Fuels and additives designated and dates prescribed by the Administrator for the registration of such fuels and additives, pursuant to section 211 of the Act, are listed in this subpart. In addition, specific informational requirements under §§ 79.11(f) and 79.21(e) are set forth for each designated fuel or additive. Additional fuels and/or additives may be designated and pertinent dates and additional specific informational requirements prescribed as the Administrator deems advisable.

§ 79.31 Additives.

(a) All additives produced or sold for use in motor vehicle gasoline and/or motor vehicle diesel fuel are hereby designated. The Act defines the term "motor vehicle" to mean any self-propelled vehicle designed for transporting persons or property on a street or highway. For purposes of this registration, however, additives specifically manufactured and marketed for use in motorcycle fuels are excluded.

(b) All designated additives must be registered by July 7, 1976, except as provided in § 79.23(b).

(c) In accordance with §§ 79.5(b) and 79.21(e), and to the extent such information is known to the additive manufacturer as a result of testing conducted for reasons other than additive registration or reporting purposes, the additive manufacturer shall furnish the highest, lowest, and average values of

the impurities in each designated additive, if greater than 0.1 percent by weight. The methods of analysis in making the determinations shall also be given.

(d) In accordance with §§ 79.5(b) and 79.21(e), and to the extent such information is known to the additive manufacturer, he shall furnish summaries of any information developed by or specifically for him concerning the following items:

(1) Mechanisms of action of the additive;

(2) Reactions between the additive and the fuels listed in paragraph (a) of this section;

(3) Identification and measurement of the emission products of the additive when used in the fuels listed in paragraph (a) of this section;

(4) Effects of the additive on all emissions;

(5) Toxicity and any other public health or welfare effects of the emission products of the additive;

(6) Effects of the emission products of the additive on the performance of emission control devices/systems. Such submissions shall be accompanied by a description of the test procedures used in obtaining the information. Information will be considered to be known to the additive manufacturer if a report thereon has been prepared and circulated or distributed outside the research department or division.

(Secs. 211, 301(a), Clean Air Act as amended (40 U.S.C. 7545, 7601(a)))

[40 FR 52011, Nov. 7, 1975, as amended at 41 FR 21324, May 25, 1976; 43 FR 28490, June 30, 1978]

§ 79.32 Motor vehicle gasoline.

(a) The following fuels commonly or commercially known or sold as motor vehicle gasoline are hereby individually designated:

(1) Motor vehicle gasoline, unleaded—motor vehicle gasoline that contains no more than 0.05 gram of lead per gallon;

(2) Motor vehicle gasoline, leaded, premium—motor vehicle gasoline that contains more than 0.05 gram of lead per gallon and is sold as "premium;"

(3) Motor vehicle gasoline, leaded, non-premium—motor vehicle gasoline

that contains more than 0.05 gram of lead per gallon but is not sold as "premium."

The Act defines the term "motor vehicle" to mean any self-propelled vehicle designed for transporting persons or property on a street or highway. For purposes of this registration, however, gasoline specifically blended and marketed for motorcycles is excluded.

(b) All designated motor vehicle gasolines must be registered by September 7, 1976.

(c) In accordance with §§ 79.5(a)(2) and 79.11(f), and to the extent such information is known to the fuel manufacturer as a result of testing conducted for reasons other than fuel registration or reporting purposes, the fuel manufacturer shall furnish the data listed below. The highest, lowest, and average values of the listed characteristics/properties are to be reported. For initial registration, data shall be given for any 3-month or longer period prior to the date of submission. For annual reports thereafter, data shall be for the calendar year, except that if the first required annual report covers a period of less than a year, the data may be for such shorter period.

(1) Hydrocarbon composition (aromatic content, olefin content, saturate content), with the methods of analysis identified;

(2) Polynuclear organic material content, sulfur content, and trace element content, with the methods of analysis identified;

(3) Reid vapor pressure;

(4) Distillation temperatures (10 percent point, end point);

(5) Research octane number and motor octane number.

(d) In accordance with §§ 79.5(a)(2) and 79.11(f), and to the extent such information is known to the fuel manufacturer, he shall furnish summaries of any information developed by or specifically for him concerning the following items:

(1) Mechanisms of action of each additive he reports;

(2) Reactions between such additives and motor vehicle gasoline;

(3) Identification and measurement of the emission products of such additives.

tives when used in motor vehicle gasoline;

(4) Effects of such additives on all emissions;

(5) Toxicity and any other public health or welfare effects of the emission products of such additives;

(6) Effects of the emission products of such additives on the performance of emission control devices/systems. Such submissions shall be accompanied by a description of the test procedures used in obtaining the information. Information will be considered to be known to the fuel manufacturer if a report thereon has been prepared and circulated or distributed outside the research department or division.

[40 FR 52011, Nov. 7, 1975, as amended at 41 FR 21324, May 25, 1976]

§ 79.33 Motor vehicle diesel fuel.

(a) The following fuels commonly or commercially known or sold as motor vehicle diesel fuel are hereby individually designated:

(1) Motor vehicle diesel fuel, grade 1-D;

(2) Motor vehicle diesel fuel, grade 2-D.

The Act defines the term "motor vehicle" to mean any self-propelled vehicle designed for transporting persons or property on a street or highway.

(b) All designated motor vehicle diesel fuels must be registered within 12 months after promulgation of this part.

(c) In accordance with §§ 79.5(a)(2) and 79.11(f), and to the extent such information is known to the fuel manufacturer as a result of testing conducted for reasons other than fuel registration or reporting purposes, the fuel manufacturer shall furnish the data listed below. The highest, lowest, and average values of the listed characteristics/properties are to be reported. For initial registration, data shall be given for any 3-month or longer period prior to the date of submission. For annual reports thereafter, data shall be for the calendar year, except that if the first required annual report covers a period of less than a year, the data may be for such shorter period.

(1) Hydrocarbon composition (aromatic content, olefin content, saturate

content), with the methods of analysis identified;

(2) Polynuclear organic material content, sulfur content, and trace element content, with the methods of analysis identified;

(3) Distillation temperatures (90 percent point, end point);

(4) Cetane number or cetane index;

(d) In accordance with §§ 79.5(a)(2) and 79.11(f), and to the extent such information is known to the fuel manufacturer, he shall furnish summaries of any information developed by or specifically for him concerning the following items:

(1) Mechanisms of action of each additive he reports;

(2) Reactions between such additives and motor vehicle diesel fuel;

(3) Identification and measurement of the emission products of such additives when used in motor vehicle diesel fuel;

(4) Effects of such additives on all emissions;

(5) Toxicity and any other public health or welfare effects of the emission products of such additives.

Such submission shall be accompanied by a description of the test procedures used in obtaining the information. Information will be considered to be known to the fuel manufacturer if a report thereon has been prepared and circulated or distributed outside the research department or division.

PART 80—REGULATION OF FUELS AND FUEL ADDITIVES

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APPENDIX F—TEST FOR DETERMINING THE QUANTITY OF ALCOHOL IN GASOLINE

AUTHORITY: Secs. 114, 211 and 301(a) of the Clean Air Act as amended, 42 U.S.C. 7414, 7545, and 7601(a).

SOURCE: 38 FR 1255, Jan. 10, 1973, unless otherwise noted.

Subpart A—General Provisions

§ 80.1 Scope.

(a) This part prescribes regulations for the control and/or prohibition of fuels and additives for use in motor vehicles and motor vehicle engines. These regulations are based upon a determination by the Administrator that the emission product of a fuel or additive will endanger the public health, or will impair to a significant degree the performance of a motor vehicle emission control device in general use or which the Administrator finds has been developed to a point where in a reasonable time it would be in general use were such regulations promulgated; and certain other findings specified by the Act.

(b) Nothing in this part is intended to preempt the ability of State or local governments to control or prohibit any fuel or additive for use in motor vehicles and motor vehicle engines which is not explicitly regulated by this part.

[38 FR 1255, Jan. 10, 1973, as amended at 42 FR 33741, Dec. 6, 1973; 42 FR 25732, Mar. 19, 1977]

§ 80.2 Definitions.

As used in this part:

(a) "Act" means the Clean Air Act as amended (42 U.S.C. 1857 et seq.).

(b) "Administrator" means the Administrator of the Environmental Protection Agency.

(c) "Gasoline" means any fuel sold in any State¹ for use in motor vehicles and motor vehicle engines, and commonly or commercially known or sold as gasoline.

(d) "Octane number, (R+M)/2 method" means measurement of a gasoline's antiknock characteristics which is obtained by dividing the sum of the Research Octane Number and the Motor Octane Number by two, as explained by the American Society for Testing and Materials (ASTM) in ASTM D 439-81, entitled "Standard Specifications for Automotive Gasoline." The Research Octane Number is determined by ASTM standard test method D 2699-80 and the Motor Octane Number is determined by ASTM standard test method D 2700-81. ASTM standards D 439-81, D 2699-80 and D 2700-81 are incorporated by reference. They are available from ASTM, 1916 Race Street, Philadelphia, PA, 19103, and are also available for inspection as part of Docket EN-81-11, located at the Central Docket Section, EPA, Gallery I, West Tower, 401 M Street, SW., Washington, DC 20460. Standard D 439-81 is contained in the Annual Book of ASTM Standards, Part 23; standards D 2699-80 and D 2700-81 are contained in Part 41. This incorporation by reference was approved by the Director of the Federal Register on January 19, 1983. These materials are incorporated as they exist on the date of the approval and a notice of any change in these materials will be published in the FEDERAL REGISTER.

¹"State" means a State, the District of Columbia, the Commonwealth of Puerto Rico, the Virgin Islands, Guam, and American Samoa.

(e) "Lead additive" means any substance containing lead or lead compounds.

(f) "Leaded gasoline" means gasoline which is produced with the use of any lead additive or which contains more than 0.05 gram of lead per gallon or more than 0.005 gram of phosphorus per gallon.

(g) "Unleaded gasoline" means gasoline which is produced without the use of any lead additive and which contains not more than 0.05 gram of lead per gallon and not more than 0.005 gram of phosphorus per gallon.

(h) "Refinery" means a plant at which gasoline is produced.

(i) "Refiner" means any person who owns, leases, operates, controls, or supervises a refinery.

(j) "Retail outlet" means any establishment at which gasoline is sold or offered for sale for use in motor vehicles.

(k) "Retailer" means any person who owns, leases, operates, controls, or supervises a retail outlet.

(l) "Distributor" means any person who transports or stores or causes the transportation or storage of gasoline at any point between any gasoline refinery or importer's facility and any retail outlet or wholesale purchaser-consumer's facility.

(m) "Lead additive manufacturer" means any person who produces a lead additive or sells a lead additive under his own name.

(n) "Reseller" means any person who purchases gasoline identified by the corporate, trade, or brand name of a refiner from such refiner or a distributor and resells or transfers it to retailers or wholesale purchaser-consumers displaying the refiner's brand, and whose assets or facilities are not substantially owned, leased, or controlled by such refiner.

(o) "Wholesale purchaser-consumer" means any organization that is an ultimate consumer of gasoline and which purchases or obtains gasoline from a supplier for use in motor vehicles and receives delivery of that product into a storage tank of at least 550-gallon capacity substantially under the control of that organization.

(p)-(q) [Reserved]

(r) "Importer" means a person who imports gasoline or gasoline blending stocks or components from a foreign country into the United States (including the Commonwealth of Puerto Rico, the Virgin Islands, Guam, American Samoa, and the Northern Mariana Islands).

(s) "Gasoline blending stock or component" means any liquid compound which is blended with other liquid compounds or with lead additives to produce gasoline.

(t) "Carrier" means any distributor who transports or stores or causes the transportation or storage of gasoline without taking title to or otherwise having any ownership of the gasoline, and without altering either the quality or quantity of the gasoline.

(u) "Ethanol blending plant" means any refinery at which gasoline is produced solely through the addition of ethanol to gasoline, and at which the quality or quantity of gasoline is not altered in any other manner.

(v) "Ethanol blender" means any person who owns, leases, operates, controls, or supervises an ethanol blending plant.

(Sec. 211 (Sec. 223, Pub. L. 95-95, 91 Stat. 764, 42 U.S.C. 7545(g)) and sec. 301(a) (42 U.S.C. 7602(a), formerly 42 U.S.C. 1857g(a)) of the Clean Air Act as amended)

[38 FR 1255, Jan. 10, 1973, as amended at 38 FR 33741, Dec. 6, 1973; 39 FR 43223 Dec. 12, 1974; 44 FR 46277, Aug. 7, 1979; 47 FR 49331, Oct. 29, 1982; 48 FR 4287, Jan. 31, 1983; 50 FR 9397, Mar. 7, 1985; 54 FR 11883, Mar. 22, 1989]

§ 80.3 Test methods.

The lead and phosphorus content of gasoline shall be determined in accordance with test methods set forth in the Appendices to this part.

[47 FR 765, Jan. 7, 1982]

§ 80.4 Right of entry; tests and inspections.

The Administrator or his authorized representative, upon presentation of appropriate credentials, shall have a right to enter upon or through any refinery, retail outlet, wholesale purchaser-consumer facility, the premises or property of any distributor or importer, or any place where gasoline is

stored, and shall have the right to make inspections, take samples and conduct tests to determine compliance with the requirements of this part.

[50 FR 9397, Mar. 7, 1985]

§ 80.5 Penalties.

Any person who violates these regulations shall forfeit and pay to the United States a civil penalty of \$10,000 for each and every day of the continuance of such violation, which shall accrue to the United States and be recovered in a civil suit in the name of the United States, brought in the district where such person has his principal office or any district in which he does business. The Administrator may, upon application by the person against whom any such penalty has been assessed, remit or mitigate any such forfeiture. The Administrator shall have authority to determine the facts upon all such applications.

§ 80.7 Requests for information.

(a) When the Administrator, the Regional Administrator, or their delegates have reason to believe a violation of section 211(c) of the Act and the regulations thereunder has occurred, they may require any refiner, distributor, wholesale purchaser-consumer, or retailer to report the following information regarding receipt, transfer, delivery, or sale of gasoline represented to be unleaded gasoline and to allow the reproduction of such information at all reasonable times.

(1) For any bulk shipment of gasoline represented to be unleaded gasoline which is transferred, sold, or delivered within the previous 6 months by a refiner or a distributor to a distributor, wholesale purchaser-consumer or a retail outlet, the refiner or distributor shall maintain and provide the following information as applicable:

(i) Business or corporate name and address of distributors, wholesale purchaser-consumers or retail outlets to which the gasoline has been transferred, sold, or delivered.

(ii) Quantity of gasoline involved.

(iii) Date of delivery.

(iv) Storage location of gasoline prior to transit via delivery vessel (e.g., location of a bulk terminal).

(v) Business or corporate name and address of the person who delivered the gasoline.

(vi) Identification of delivery vessel (e.g., truck number). This information shall be supplied by the person performing the delivery, e.g., common carrier or contract carrier.

(2) For any bulk shipment of gasoline represented to be unleaded gasoline received by a retail outlet or wholesale-purchaser-consumer facility within the previous 6 months, whether by purchase or otherwise, the retailer or wholesale purchaser-consumer shall maintain accessibility to and provide the following information:

(i) Business or corporate name and address of the distributor.

(ii) Quantity of gasoline received.

(iii) Date of receipt.

(b) Upon request by the Administrator, the Regional Administrator, or their delegates, any retailer shall provide documentation of his annual total sales volume in gallons of gasoline for each retail outlet for each calendar year beginning with 1971.

(c) Any refiner, distributor, wholesale purchaser-consumer, retailer, or importer shall provide such other information as the Administrator or his authorized representative may reasonably require to enable him to determine whether such refiner, distributor, wholesale purchaser-consumer, retailer, or importer has acted or is acting in compliance with section 211(c) of the Act and the regulations thereunder and shall, upon the request of the Administrator or his authorized representative, produce and allow reproduction of any relevant records at all reasonable times. Such information may include but is not limited to records of unleaded gasoline inventory at a wholesale purchaser-consumer facility or a retail outlet, unleaded pump meter readings at a wholesale purchaser-consumer facility or a retail outlet, and receipts providing the date of acquisition of signs, labels, and nozzles required by § 80.22. No person shall be required to furnish information requested under this paragraph if he can establish that such information is not maintained in the normal course of his business.

(Secs. 211, 301, Clean Air Act as amended (42 U.S.C. 1857f-6c, 1857g))

(40 FR 36336, Aug. 20, 1975, as amended at 42 FR 45307, Sept. 9, 1977; 47 FR 49332, Oct. 29, 1982)

Subpart B—Controls and Prohibitions

§ 80.20 Controls applicable to gasoline refiners and importers.

(a) *Refiners.* (1) In the production of gasoline at a refinery, a refiner shall not:

(i) Produce leaded gasoline whose average lead content during any calendar quarter ending prior to July 1, 1985, exceeds 1.10 grams of lead per gallon of leaded gasoline.

(ii) Produce leaded gasoline whose average lead content during any calendar quarter beginning on or after July 1, 1985, and ending prior to January 1, 1986, exceeds 0.50 gram of lead per gallon of leaded gasoline.

(iii) Produce leaded gasoline whose average lead content during any calendar quarter beginning on or after January 1, 1986, exceeds 0.10 gram of lead per gallon of leaded gasoline.

(2) Except as provided in paragraph (d)(1) of this section, compliance with the requirements of paragraph (a)(1)(i), (ii) and (iii) of this section shall be determined by dividing the total grams of lead used in the production of leaded gasoline (including the lead in gasoline blending stocks and components used in such production) at a refinery during a calendar quarter by the total gallons of leaded gasoline produced at the refinery in the same calendar quarter.

(3) For each calendar quarter, each refiner shall submit to the Administrator a report which contains the following information for each refinery:

(i) The total grams of lead in the refinery's inventory (including its lead additive inventory and its inventory of gasoline blending stocks and components) on the first day of the calendar quarter;

(ii) The total grams of lead (including lead additives and lead in gasoline blending stocks and components) received by the refinery during the calendar quarter;

(iii) The total grams of lead additives shipped from the refinery during the calendar quarter;

(iv) The total grams of lead in the refinery's inventory (including its lead additive inventory and its inventory of gasoline blending stocks and components) on the last day of the calendar quarter;

(v) The total gallons of leaded gasoline produced by the refinery during the calendar quarter;

(vi) The total gallons of unleaded gasoline produced by the refinery during the calendar quarter;

(vii) The total grams of lead used in the production of leaded gasoline (including lead additives and the lead in gasoline blending stocks and components used in such production) by the refinery during the calendar quarter;

(viii) The average lead content of each gallon of leaded gasoline produced by the refinery during the calendar quarter;

(ix) The total grams of lead used in the production of products other than gasoline by the refinery during the calendar quarter, by type of product;

(x) The total gallons of products other than gasoline in which lead was used that were produced by the refinery during the calendar quarter, by type of product; and

(xi) If any of the products listed in paragraph (a)(3)(x) were sold or otherwise transferred to another refinery during the calendar quarter, the total gallons of each product so transferred, the total grams of lead in each product so transferred, the name and address of the refinery to which the transfer was made, and the date of such transfer.

Reports shall be submitted within 15 days after the close of the calendar quarter on forms prescribed by the Administrator.

(b) [Reserved]

(c) *Importers.* (1)(i) No importer shall sell or offer for sale leaded gasoline which has been imported into the United States and whose average lead content during any calendar quarter ending prior to July 1, 1985, exceeds 1.10 grams of lead per gallon of such gasoline.

(ii) No importer shall sell or offer for sale leaded gasoline which has been

imported into the United States and whose average lead content during any calendar quarter beginning on or after July 1, 1985, and ending prior to January 1, 1986, exceeds 0.50 gram of lead per gallon of such gasoline.

(iii) No importer shall sell or offer for sale leaded gasoline whose average lead content during any calendar quarter beginning on or after January 1, 1986, exceeds 0.10 grams of lead per gallon of such gasoline.

(2) Except as provided in paragraph (d)(1) of this section, compliance with the requirements of paragraphs (c)(1)(i), (ii), and (iii) shall be determined by calculating:

(i) The lead content of each shipment of imported leaded gasoline sold by the importer during a calendar quarter, determined by the performance by the importer of the test for lead in gasoline set forth in Appendix B of this part upon a representative sample of gasoline in the shipment;

(ii) The total gallons of leaded gasoline in each such shipment;

(iii) The total grams of lead in each such shipment, determined by multiplying the lead content of the shipment by the total gallons of leaded gasoline in the shipment;

(iv) The total grams of lead in all such shipments sold during the calendar quarter;

(v) The total gallons of leaded gasoline in all such shipments sold during the calendar quarter;

(vi) The average lead content of all imported leaded gasoline sold during the calendar quarter, determined by dividing the total in paragraph (c)(2)(iv) by the total in paragraph (c)(2)(v).

(3) For each calendar quarter, each importer who sells imported leaded gasoline or imported gasoline blending stocks or components shall submit to the Administrator a report which contains the following information:

(i) The information described in paragraphs (c)(2) (i) through (vi) of this section;

(ii) The lead content of each shipment of imported gasoline blending stocks or components sold by the importer during the calendar quarter, determined by performance by the importer of the test for lead in gasoline

set forth in Appendix B of this Part upon a representative sample of gasoline blending stocks or components in the shipment;

(iii) The total gallons of gasoline blending stocks or components in each such shipment;

(iv) The total grams of lead in each such shipment, determined by multiplying the lead content of the shipment by the total gallons of gasoline blending stocks or components in the shipment;

(v) For each shipment of imported leaded gasoline or imported gasoline blending stocks or components sold during the calendar quarter: name and address of importer; date and place of entry; and vessel or carrier number (where applicable); and

(vi) For each shipment of imported leaded gasoline blending stocks or components sold during the calendar quarter: the name and address of the refinery or the other person to which the sale was made; the total gallons of product sold; the total grams of lead in the product sold; and the date of such sale.

Reports shall be submitted within 15 calendar days after the close of the calendar quarter on forms prescribed by the Administrator.

(4) Any importer who adds lead to gasoline or gasoline blending stocks or components during a calendar quarter shall also submit a report pursuant to paragraph (a)(3) of this section.

(d) *Inter-refinery averaging.* (1) As an alternative means of demonstrating compliance with the requirements of paragraph (a)(1)(i), (a)(1)(ii), (c)(1)(i), or (c)(1)(ii) of this section, one or more refiners may demonstrate such compliance by constructively allocating lead usage between or among two or more refineries in any manner agreed upon by the refiner(s), so long as:

(i) The average constructive lead content of leaded gasoline produced in a calendar quarter by each refinery does not exceed the lead content standard applicable to such calendar quarter (as prescribed in paragraph (a)(1)(i), (a)(1)(ii), (c)(1)(i), or (c)(1)(ii) of this section);

(ii) The total amount of lead usage in a calendar quarter by all such refin-

eries, as constructively allocated and reported, is equal to the total amount of lead actually used in the calendar quarter by all such refineries;

(iii) The actual or constructive lead content of gasoline produced by each refinery does not exceed any applicable state statutory or regulatory standards; and

(iv) The constructive allocation agreement is made no later than the final day of the calendar quarter in which the lead allocated is actually used.

(2) Any refiner who demonstrates compliance with the requirements of this section pursuant to paragraph (d)(1) of this section shall submit to the Administrator, as an additional part of the report required by paragraph (a)(3) or paragraph (c)(3) of this section, the following information:

(i) The total grams of lead actually used by the reporting refinery during the calendar quarter and constructively allocated to another refinery, and the name and address of such other refinery (for each such constructive allocation);

(ii) The total grams of lead actually used by another refinery during the calendar quarter and constructively allocated to the reporting refinery, and the name and address of such other refinery (for each such constructive allocation);

(iii) The total grams of lead constructively used in the production of leaded gasoline by the reporting refinery during the calendar quarter, as determined by performing the following calculations upon the total grams of lead actually used by the reporting refinery during the calendar quarter: (A) Subtracting the total grams of lead indicated in paragraph (d)(2)(i) of this section, and (B) Adding the total grams of lead indicated in paragraph (d)(2)(ii) of this section; and

(iv) The constructive average lead content of leaded gasoline produced by the reporting refinery during the calendar quarter, as determined by dividing the total grams of lead indicated in paragraph (d)(2)(iii) of this section by the total gallons of leaded gasoline produced by the reporting refinery during the calendar quarter; and

(v) When compliance is demonstrated pursuant to paragraph (d)(1) by more than one refiner, each such report shall also include supporting documentation adequate to show the agreement of all such refiners to the constructive allocation of lead usage stated in the report.

(3) For purposes of paragraphs (d)(1) and (d)(2) of this section, the total amount of imported leaded gasoline sold during a calendar quarter by each importer shall be treated as the output of a single refinery, and each importer shall be treated as a refiner.

(4) The provisions of paragraphs (d)(1), (d)(2), and (d)(3) of this section shall not be applicable during any calendar quarter beginning on or after January 1, 1986.

(e) *Banking and withdrawal of lead usage rights*—(1) *Banking of lead usage rights.* (i) During any calendar quarter beginning on or after January 1, 1985, and ending prior to January 1, 1986, a refiner may bank lead usage rights in an amount equal to the amount of lead usage allowed at a refinery in the calendar quarter minus the amount of lead actually or constructively used in the same calendar quarter at the refinery, as determined under paragraphs (e)(1) (ii) and (iii) of this section.

(ii) The amount of lead usage rights that may be banked by a refiner at a refinery pursuant to paragraph (e)(1)(i) of this section shall be equal to the number of grams of lead determined by:

(A) Multiplying the number of gallons of leaded gasoline reported by the refinery for the calendar quarter pursuant to paragraph (a)(3)(v) or (c)(2)(v) of this section by the Federal lead content standard applicable to the refinery during the calendar quarter (as set forth in paragraph (a)(1)(i), (a)(1)(ii), (c)(1)(i), or (c)(1)(ii) of this section) or any applicable state lead content standard prescribed pursuant to section 211(c) (4) (B) or (C) of the Clean Air Act, whichever allows less lead usage; and

(B) Subtracting from the result in paragraph (e)(1)(ii)(A) the total grams of lead reported pursuant to paragraph (a)(3)(vii) or (c)(2)(iv) of this section.

(iii) When compliance with the requirements of paragraph (a)(1)(i), (a)(1)(ii), (c)(1)(i) or (c)(1)(ii) is demonstrated pursuant to the inter-refinery averaging provisions of paragraph (d) of this section, the total grams of lead reported pursuant to paragraph (d)(2)(iii) of this section shall be used instead of the total grams of lead reported pursuant to paragraph (a)(3)(vii) or (c)(2)(iv) of this section, for purposes of paragraph (e)(1)(ii)(B) of this section.

(iv) the total grams of lead used for purposes of paragraph (e)(1)(ii)(B) of this section may not be less than the amount calculated by multiplying the number of gallons of leaded gasoline reported by the refinery for the calendar quarter pursuant to paragraph (a)(3)(v) or (c)(2)(v) of this section by 0.10 gram of lead per gallon of such gasoline.

(v) A refinery who banks lead usage rights pursuant to this sub-section shall submit to the Administrator, as an additional part of the report required by paragraph (a)(3) or (c)(3) of this section, the information described in paragraph (e)(1)(ii) and (iii) of this section.

(2) *Withdrawal of lead usage rights.*

(i) During any calendar quarter beginning on or after April 1, 1985, and ending prior to January 1, 1988, a refinery may withdraw lead usage rights banked pursuant to paragraph (e)(1) of this section. Such rights may be used by the refinery to demonstrate compliance with the requirements of paragraph (a)(1) or (c)(1), or may be transferred by the refinery for such use by another refinery. A lead usage right shall be considered to be withdrawn when it is so used or transferred by the banking refinery.

(ii) Compliance with the requirements of paragraph (a)(1) or (c)(1) through the use of lead usage rights shall be determined by:

(A) Subtracting the lead usage rights used by the reporting refinery from the total grams of lead reported pursuant to paragraph (a)(3)(vii) or (c)(2)(iv) of this section (or, if compliance is also demonstrated pursuant to the inter-refinery averaging provisions of paragraph (d) of this section, subtracting the lead usage rights used by

the reporting refinery from the total grams of lead reported pursuant to paragraph (d)(2)(iii) of this section); and

(B) Dividing the result in paragraph (e)(2)(ii)(A) by the number of gallons of leaded gasoline reported pursuant to paragraph (a)(3)(v) or (c)(2)(v) of this section.

(iii) A refinery who deposits, withdraws, transfers, or uses lead usage rights during a calendar quarter shall submit to the Administrator, as an additional part of the report required by paragraph (a)(3) or (c)(3) of this section, the following information:

(A) The amount of lead usage rights withdrawn from the reporting refinery's account and used to demonstrate compliance with the requirements of paragraph (a)(1) or (c)(1) for the calendar quarter;

(B) The amount of lead usage rights withdrawn from the reporting refinery's account and transferred to the account of another refinery during the calendar quarter;

(C) The amount of lead usage rights transferred from the account of another refinery to that of the reporting refinery and used to demonstrate compliance with the requirements of paragraph (a)(1) or (c)(1) for the calendar quarter;

(D) The amount of lead usage rights transferred from the account of another refinery in the calendar quarter and deposited in the account of the reporting refinery during the calendar quarter;

(E) The banked average lead content of leaded gasoline produced in the calendar quarter, as determined pursuant to paragraph (e)(2)(ii) of this section;

(F) If the other refinery to or from which the lead usage rights reported in paragraph (e)(2)(iii) (B), (C) or (D) were transferred is owned or controlled by another refinery, supporting documentation adequate to show the agreement by such refinery to the transfer of the lead usage rights.

(3) *Other requirements.* (i) For purposes of paragraph (e) of this section, the total amount of imported leaded gasoline sold during a calendar quarter by each importer shall be treated as the output of a single refinery, and

each importer shall be treated as a refiner.

(ii) The banked average lead content of leaded gasoline produced at a refinery during a calendar quarter, as determined pursuant to paragraph (e)(2)(ii) of this section, may not exceed any applicable state gasoline lead content standards prescribed pursuant to section 211(c)(4) (B) or (C) of the Clean Air Act.

(iii) An agreement between two (or more) refiners to transfer lead usage rights may be made no later than the final day of the calendar quarter in which the lead usage rights are used.

(iv) Any refiner who has lead usage rights on deposit in a lead usage rights bank account at the end of a calendar quarter or who makes deposits to or withdrawals from a lead usage rights banking account under the provisions of paragraph (e) of this section during a calendar quarter shall submit to the Administrator, as an additional part of the report required by paragraph (a)(3) or (c)(3) of this section, the following information:

(A) The number of grams of lead usage rights in the refinery's lead usage rights bank account at the beginning of the calendar quarter for which the report is submitted;

(B) The number of grams of lead usage rights in the refinery's lead usage rights bank account at the close of the calendar quarter for which the report is submitted (after all deposits and withdrawals in such account during the calendar quarter have been taken into account).

[50 FR 9397, Mar. 7, 1985 and 50 FR 13128, Apr. 2, 1985; 50 FR 25710, June 21, 1985]

§ 80.21 Controls applicable to gasoline distributors.

(a) After July 1, 1974 no distributor shall sell or transfer to any distributor, retailer or wholesale purchaser-consumer any gasoline which he represents is unleaded unless such gasoline does, in fact, meet the defined requirements for unleaded gasoline in § 80.2(g).

(b) No carrier or his employee or agent, whether operating under contract or tariff, shall cause unleaded gasoline tendered to the carrier for shipment or transfer to another carrier,

distributor, or retailer to fail to comply, at the time of delivery, with the defined requirements for unleaded gasoline in § 80.2(g).

[39 FR 43283, Dec. 12, 1974, as amended at 40 FR 29292, July 11, 1975]

§ 80.22 Controls applicable to gasoline retailers and wholesale purchaser-consumers.

(a) After July 1, 1974 no retailer or his employee or agent and after January 31, 1975 no wholesale purchaser-consumer or his employee or agent shall sell, dispense, or offer for sale gasoline represented to be unleaded unless such gasoline meets the defined requirements for unleaded gasoline in § 80.2(g); nor shall he introduce, or cause or allow the introduction of leaded gasoline into any motor vehicle which is labeled "unleaded gasoline only," or which is equipped with a gasoline tank filler inlet which is designed for the introduction of unleaded gasoline.

(b) After July 1, 1974 every person who owns, leases, operates, controls or supervises one or more retail outlets with gasoline sales as described in paragraph (b)(1)(i) of this section or with gasoline sales and location as described in paragraph (b)(1)(ii) of this section shall offer for sale at each such retail outlet at least one grade of unleaded gasoline of not less than 87 Octane, (R+M)/2 method: *Provided, however,* That the octane number of unleaded gasoline may be reduced for altitude as specified in American Society for Testing and Materials Standard D 439-81, which is incorporated by reference (see 40 CFR 80.2 (d)), except that references in that Standard to EPA regulations affecting Research Octane Number (RON) shall not apply.

(1) Unleaded gasoline must be offered for sale at each retail outlet:

(i) At which 200,000 or more gallons of gasoline were sold during any calendar year beginning with the year 1971;

(ii) At which 150,000 or more gallons of gasoline were sold during any calendar year beginning with the year 1971 if such retail outlet is located in a county containing a population density of less than 50 persons per square

mile according to statistical data from the 1970 census of population and calculated by excluding cities² containing populations of 50,000 or more persons, unless such retail outlet is located in a city of 10,000 or more persons.

(2) Any retailer may defer compliance with paragraph (b)(1)(ii) of this section until January 31, 1975. However, if a retailer subject to paragraph (b)(1)(ii) of this section offers for sale unleaded gasoline at such outlet prior to that date, the retailer shall comply with all applicable provisions of these regulations.

(c) Time extensions or exemptions to these regulations may be granted upon conformance with the following conditions: (1) Any retailer may obtain an exemption from paragraph (b) of this section for outlets at which business will be terminated by January 1, 1975. In order to obtain this exemption, the retailer must present evidence to the Regional Administrator of specific termination plans for each such outlet. Any retailer granted this exemption for a specified outlet(s) who offers for sale unleaded gasoline at such outlet after July 1, 1974, but before January 1, 1975, shall inform the Regional Administrator in writing of such an offering and shall be expected to comply with all applicable provisions of these regulations.

(2) Any retailer may obtain a time extension for compliance with paragraph (b) of this section until September 1, 1974, for outlets at which equipment procurement delays will preclude compliance with these regulations. The extension is limited to a retailer who can present evidence to the Regional Administrator, upon his request, that the equipment was ordered in a timely manner but delivery was delayed for reasons beyond the control of the retail outlet owner and an attempt to procure equipment from alternative sources was made promptly by the owner upon receipt of notice of the delay.

²"City" means a political subdivision of a State within a defined area over which a municipal corporation has been established to provide local government functions and facilities.

(3) The Regional Administrator or his authorized representative may grant a time extension to any retailer to defer compliance with paragraph (b)(1)(ii) of this section upon a showing that

(i) Compliance with this section within that period of time would result in an inability to continue in business or would result in severe economic hardship.

(ii) Additional time is necessary to procure a nozzle spout as described in § 80.22(f)(2), or

(iii) Additional time is necessary to prepare existing storage tanks to dispense unleaded gasoline and to offer unleaded gasoline for sale.

Time extensions under paragraph (c)(3)(i) may be granted for a period of time of up to six months. Time extensions under paragraphs (c)(3)(ii) and (iii) may be granted for a period of time of up to two months. Any extension under paragraph (c)(3) may be renewed by the Regional Administrator or his authorized representative upon a showing that the retailer continues to qualify for an extension.

(4) All applications for exemptions or time extensions under paragraphs (c)(1), (c)(2) and (c)(3) of this section shall state the name and address of the applicant and address of the retail outlet and shall be sent to the appropriate Regional Administrator, or his authorized representative, of the Environmental Protection Agency in the EPA region in which the retail outlet is located. Time extensions to the regulations pursuant to paragraph (c)(3) may be granted upon conformance with the following conditions:

(i) Any retailer requesting a time extension under paragraph (c)(3)(i) of this section shall contact the Regional Administrator or his authorized representative in his region who will inform the applicant of the documentation required to support his request for an extension.

(ii) Any retailer requesting a time extension under paragraph (c)(3)(ii) of this section shall present to the Regional Administrator or his authorized representative, upon his request, evidence that the equipment was ordered in a timely manner but delivery was delayed for reasons beyond the control

of the retailer, and an attempt to procure equipment from an alternative source was made promptly and continues to be made by the retailer upon receipt of the notice of the delay.

(iii) Any retailer requesting a time extension under paragraph (c)(3)(iii) of this section shall present to the Regional Administrator or his authorized representative, upon his request, evidence that unleaded gasoline was ordered in a timely manner but delivery was delayed for reasons beyond the control of the retailer, and an attempt to procure unleaded gasoline from an alternative source was made promptly by the retailer upon receipt of notice of the delay, and continues to be made (including an active application for the assignment of a supplier on file with his regional Federal Energy Administration Office).

A listing of the addresses of the Environmental Protection Agency regional offices follows:

I. John F. Kennedy Federal Building, Room 2203, Boston, Massachusetts 02203.

II. 26 Federal Plaza, Room 908, New York, New York 10007.

III. Curtis Building, 6th & Walnut Streets, Philadelphia, Pennsylvania 19106.

IV. 1421 Peachtree Street, N.E., Atlanta, Georgia 30309.

V. 230 South Dearborn Street, Chicago, Illinois.

VI. 1600 Patterson Street, Suite 1100, Dallas, Texas 75201.

VII. 1735 Baltimore Avenue, Kansas City, Missouri 64108.

VIII. 1860 Lincoln Street, Suite 900, Denver, Colorado 80203.

IX. 100 California Street, San Francisco, California 94111.

X. 1200 6th Avenue, Seattle, Washington 98101.

(d) After July 1, 1974 every retailer shall prominently and conspicuously display in the immediate area of each gasoline pump stand, and after January 31, 1975 every wholesale purchaser-consumer shall prominently and conspicuously display in the immediate area of each pump stand, the following notice:

Federal law prohibits the introduction of any gasoline containing lead or phosphorus into any motor vehicle labeled "UNLEADED GASOLINE ONLY." Such notice shall be no smaller than 36-point bold type and shall be located so as to be readily visible to the retailer's or wholesale purchaser-con-

sumer's employees and persons operating vehicles into which gasoline is to be dispensed.

(e) After July 1, 1974 every retailer shall affix to each gasoline pump stand a permanent legible label, and after January 31, 1975 every wholesale purchaser-consumer shall affix to each gasoline pump stand a permanent legible label as follows:

(1) For gasoline pump stands containing pumps for introduction of unleaded gasoline into motor vehicles, the label shall state: Unleaded gasoline.

(2) For gasoline pump stands containing pumps for introduction of leaded gasoline into motor vehicles, the label shall state: Contains lead anti-knock compounds. Any label required under this paragraph shall be located so as to be readily visible to the retailer's or wholesale purchaser-consumer's employees and persons operating motor vehicles into which gasoline is to be dispensed.

(f) After July 1, 1974 every retailer shall equip all gasoline pumps, and after January 31, 1975 every wholesale purchaser-consumer shall equip all gasoline pumps as follows:

(1) Each pump from which leaded gasoline is introduced into motor vehicles shall be equipped with a nozzle spout having a terminal end with an outside diameter of not less than 0.930 inch (2.363 centimeters).

(2) Each pump from which unleaded gasoline is introduced into motor vehicles shall be equipped with a nozzle spout which meets the following specifications:

(i) The outside diameter of the terminal end shall not be greater than 0.840 inch (2.134 centimeters);

(ii) The terminal end shall have a straight section of at least 2.5 inches (6.34 centimeters) in length; and

(iii) The retaining spring shall terminate 3.0 inches (7.6 centimeters) from the terminal end.

(g) If more than one grade of gasoline is dispensed from a gasoline pump or pump stand, the Administrator may grant an exception to paragraph (e) or (f) of this section where it has been demonstrated to his satisfaction that an alternate system of labeling or

equipment will comply with the objectives of paragraph (e) or (f) of this section.

(h) If more than one grade of unleaded gasoline is offered for sale at a retail outlet covered by these regulations, compliance with paragraph (e) or (f) of this section is required for only one grade.

(i) An exemption to paragraph (b) of this section is provided for any retail outlet which is otherwise required to carry a grade of unleaded gasoline and which has sold less than 100,000 gallons of gasoline to light duty motor vehicles in the year 1971 and in each subsequent year.

[38 FR 1255, Jan. 10, 1973, as amended at 39 FR 16125, May 17, 1974; 39 FR 43283, Dec. 12, 1974; 48 FR 4287, Jan. 31, 1983]

§ 80.23 Liability for violations.

Liability for violations of paragraph (a) of § 80.22 shall be determined as follows:

(a)(1) Where the corporate, trade, or brand name of a gasoline refiner or any of its marketing subsidiaries appears on the pump stand or is displayed at the retail outlet or wholesale purchaser-consumer facility from which the gasoline was sold, dispensed, or offered for sale, the retailer or wholesale purchaser-consumer, the reseller (if any), and such gasoline refiner shall be deemed in violation. Except as provided in paragraph (b)(2) of this section, the refiner shall be deemed in violation irrespective of whether any other refiner, distributor, retailer, or wholesale purchaser-consumer or the employee or agent of any refiner, distributor, retailer, or wholesale purchaser-consumer may have caused or permitted the violation.

(2) Where the corporate, trade, or brand name of a gasoline refiner or any of its marketing subsidiaries does not appear on the pump stand and is not displayed at the retail outlet or wholesale purchaser-consumer facility from which the gasoline was sold, dispensed, or offered for sale, the retailer or wholesale purchaser-consumer and any distributor who sold that person gasoline contained in the storage tank which supplied that pump at the time of the violation shall be deemed in violation.

(b)(1) In any case in which a retailer or wholesale purchaser-consumer and any gasoline refiner or distributor would be in violation under paragraph (a) (1) or (2) of this section, the retailer or wholesale purchaser-consumer shall not be liable if he can demonstrate that the violation was not caused by him or his employee or agent.

(2) In any case in which a retailer or wholesale purchaser-consumer, a reseller (if any), and any gasoline refiner would be in violation under paragraph (a)(1) of this section, the refiner shall not be deemed in violation if he can demonstrate:

(i) That the violation was not caused by him or his employee or agent, and

(ii) That the violation was caused by an act in violation of law (other than the Act or this part), or an act of sabotage, vandalism, or deliberate commingling of leaded and unleaded gasoline, whether or not such acts are violations of law in the jurisdiction where the violation of the requirements of this part occurred, or

(iii) That the violation was caused by the action of a reseller or a retailer supplied by such reseller, in violation of a contractual undertaking imposed by the refiner on such reseller designed to prevent such action, and despite reasonable efforts by the refiner (such as periodic sampling) to insure compliance with such contractual obligation, or

(iv) That the violation was caused by the action of a retailer who is supplied directly by the refiner (and not by a reseller), in violation of a contractual undertaking imposed by the refiner on such retailer designed to prevent such action, and despite reasonable efforts by the refiner (such as periodic sampling) to insure compliance with such contractual obligation, or

(v) That the violation was caused by the action of a distributor subject to a contract with the refiner for transportation of gasoline from a terminal to a distributor, retailer or wholesale purchaser-consumer, in violation of a contractual undertaking imposed by the refiner on such distributor designed to prevent such action, and despite reasonable efforts by the refiner (such as periodic sampling) to insure compli-

ance with such contractual obligation, or

(vi) That the violation was caused by a distributor (such as a common carrier) not subject to a contract with the refiner but engaged by him for transportation of gasoline from a terminal to a distributor, retailer or wholesale purchaser-consumer, despite reasonable efforts by the refiner (such as specification or inspection of equipment) to prevent such action, or

(vii) That the violation occurred at a wholesale purchaser-consumer facility: *Provided, however,* That if such wholesale purchaser-consumer, was supplied by a reseller, the refiner must demonstrate that the violation could not have been prevented by such reseller's compliance with a contractual undertaking imposed by the refiner on such reseller as provided in paragraph (b)(2)(iii) of this section.

(viii) In paragraphs (b)(2)(ii) through (vi) hereof, the term "was caused" means that the refiner must demonstrate by reasonably specific showings by direct or circumstantial evidence that the violation was caused or must have been caused by another.

(c) In any case in which a retailer or wholesale purchaser-consumer, a reseller, and any gasoline refiner would be in violation under paragraph (a)(1) of this section, the reseller shall not be deemed in violation if he can demonstrate that the violation was not caused by him or his employee or agent.

(d) In any case in which a retailer or wholesale purchaser-consumer and any gasoline distributor would be in violation under paragraph (a)(2) of this section, the distributor will not be deemed in violation if he can demonstrate that the violation was not caused by him or his employee or agent.

(e)(1) In any case in which a retailer or his employee or agent or a wholesale purchaser-consumer or his employee or agent introduced leaded gasoline from a pump from which leaded gasoline is sold, dispensed, or offered for sale, into a motor vehicle which is equipped with a gasoline tank filler inlet designed for the introduction of unleaded gasoline, only the retailer or

wholesale purchaser-consumer shall be deemed in violation.

(2) If a retailer or a wholesale purchaser-consumer establishes that the conduct referred to in paragraph (e)(1) of this section was in response to the requirements of a *bona fide* emergency (such as when the gasoline tank of a vehicle is almost empty and no unleaded gasoline is available within a several mile radius), the retailer or wholesale purchaser-consumer will not be deemed in violation: *Provided, however,* That the amount of leaded gasoline which was introduced into the vehicle was limited to no more than was reasonably required to alleviate the circumstances of the particular emergency situation.

(Secs. 211, 301 of the Clean Air Act, as amended (42 U.S.C. 1857f-6c, 1857g))

[38 FR 1255, Jan. 10, 1973, as amended at 39 FR 42360, Dec. 5, 1974; 39 FR 43284, Dec. 12, 1974; 42 FR 45307, Sept. 9, 1977]

§ 80.24 Controls applicable to motor vehicle manufacturers.

The manufacturer of any motor vehicle equipped with an emission control device which the Administrator has determined will be significantly impaired by the use of leaded gasoline shall:

(a) Affix two or more permanent legible labels reading "Unleaded Gasoline Only" or "Unleaded Fuel Only" to such vehicle at the time of its manufacture, as follows:

(1) One label shall be located on the instrument panel so as to be readily visible to the operator of the vehicle: *Provided, however,* That the required statement may be incorporated into the design of the instrument panel rather than provided on a separate label, and that this paragraph (a)(1) shall not apply to motorcycles as defined at 40 CFR 86.402-78; and

(2) One label shall be located immediately adjacent to each gasoline filler tank inlet, outside of any filler inlet compartment, and shall be located so as to be readily visible to any person introducing gasoline to such filler inlet, and for motorcycles, such label shall be readily visible to any person introducing gasoline to such filler inlet from the left side and the right side of

the vehicle: *Provided, however*, That the Administrator may, upon application from a motor vehicle manufacturer, approve other label locations that achieve the purpose of this paragraph.

(3) Such labels shall be in the English language in block letters which shall be of a color that contrasts with their background.

(b) Manufacture such vehicle with each gasoline tank filler inlet having a restriction which prevents the insertion of a nozzle with a spout as described in § 80.22(f)(1) and allows the insertion of a nozzle with a spout as described in § 80.22(f)(2).

(1) Such filler inlet shall be designed to pass not more than 700 c.c. of gasoline into the tank when the introduction of gasoline into such filler inlet is attempted from a nozzle, as described in § 80.22(f)(1), which has a vacuum port the center of which is located within 0.87 inches of the tip and which passes less than 120 c.c. of fuel when fully and rapidly activated with the nozzle vacuum port plugged. During such attempted introduction, the nozzle shall be inserted such that its automatic shutoff vacuum port is at various depths within the filler inlet, except those locations which cause fuel spillage (not including splash back) outside the filler inlet shall not be used. The nozzle may have any orientation within the filler inlet which may reasonably be expected to be encountered in use. The nozzle valve shall be fully and rapidly opened to a 8 ± 1 gallon/minute flow setting. This test is conducted using a test fixture which positions the filler inlet pipe in the same position as it is installed in the vehicle.

(2) Paragraph (b)(1) of this section shall not apply to motorcycles.

(c) For purposes of this section a motor vehicle shall be deemed to be equipped with an emission control device which will be significantly impaired by the use of leaded gasoline if unleaded gasoline was used in any testing or service accumulation under Part 86 relating to the emission certification of said motor vehicles or engine installed therein.

[38 FR 26450, Sept. 21, 1973, as amended at 39 FR 34538, Sept. 26, 1974; 46 FR 50472,

Oct. 13, 1981; 48 FR 29692, June 28, 1983; 51 FR 33731, Sept. 22, 1986]

§ 80.25 Controls applicable to lead additive manufacturers.

For each 3-month period (January through March, April through June, July through September, October through December) commencing with the period January 1, 1975 through March 31, 1975, each lead additive manufacturer shall submit to the Administrator a report showing the total grams of lead shipped to each refinery by such lead additive manufacturer during the period. Reports shall be submitted within 15 days after the close of the reporting period, on forms supplied by the Administrator upon request.

[38 FR 33741, Dec. 6, 1973]

§ 80.26 Confidentiality of information.

Information obtained by the Administrator or his representatives pursuant to this part shall be treated, in so far as its confidentiality is concerned, in accordance with the provisions of 40 CFR Part 2.

[38 FR 33741, Dec. 6, 1973]

§ 80.27 Controls and prohibitions on gasoline volatility.

(a) *Prohibited activities.* During regulatory control periods no refiner, importer, distributor, reseller, carrier, ethanol blender, retailer or wholesale purchaser-consumer shall sell, offer for sale, dispense, supply, offer for supply, or transport gasoline whose Reid vapor pressure exceeds the applicable standard. As used in this section and § 80.28, "applicable standard" means the standard listed in this paragraph for the geographical area and time period in which gasoline is intended to be dispensed to motor vehicles or, if such area and time period cannot be determined, the standard listed in this paragraph that specifies the lowest Reid vapor pressure for the year in which the gasoline is sampled. As used in this section and § 80.28, "regulatory control periods" mean the following periods during calendar year 1989: June 30, to September 15 for retail outlets and wholesale purchaser-consumer facilities; and June 1, to

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September 15 for all other facilities. As used in this section and § 80.28, "regulatory control periods" mean the following periods during calendar year 1990 and each calendar year thereafter:

June 1 to September 15 for retail outlets and wholesale purchaser-consumer facilities; and May 1 to September 15 for all other facilities.

(1) Applicable Standards¹ 1989-1991.

APPLICABLE STANDARDS¹

State	May	June	July	Aug.	Sept.
Alabama	10.5	10.5	9.5	9.5	10.5
Arizona	9.5	9.0	9.0	9.0	9.5
Arkansas	10.5	10.5	9.5	9.5	10.5
California: ²					
North Coast	10.5	9.5	9.5	9.5	9.5
South Coast	9.5	9.5	9.5	9.5	9.5
Southeast	9.5	9.5	9.5	9.5	9.5
Interior	9.5	9.5	9.5	9.5	9.5
Colorado	10.5	9.5	9.5	9.5	9.5
Connecticut	10.5	10.5	10.5	10.5	10.5
Delaware	10.5	10.5	10.5	10.5	10.5
District of Columbia	10.5	10.5	10.5	10.5	10.5
Florida	10.5	10.5	10.5	10.5	10.5
Georgia	10.5	10.5	9.5	9.5	10.5
Idaho	10.5	10.5	10.5	10.5	10.5
Illinois					
North of 40° Latitude	10.5	10.5	10.5	10.5	10.5
South of 40° Latitude	10.5	10.5	9.5	9.5	10.5
Indiana	10.5	10.5	10.5	10.5	10.5
Iowa	10.5	10.5	10.5	10.5	10.5
Kansas	10.5	10.5	10.5	10.5	10.5
Kentucky	10.5	10.5	9.5	9.5	10.5
Louisiana	10.5	10.5	10.5	10.5	10.5
Maine	10.5	10.5	9.5	9.5	10.5
Maryland	10.5	10.5	10.5	10.5	10.5
Massachusetts	10.5	10.5	10.5	10.5	10.5
Michigan	10.5	10.5	10.5	10.5	10.5
Minnesota	10.5	10.5	10.5	10.5	10.5
Mississippi	10.5	10.5	9.5	9.5	10.5
Missouri	10.5	10.5	9.5	9.5	10.5
Montana	10.5	10.5	10.5	10.5	10.5
Nebraska	10.5	10.5	10.5	10.5	10.5
Nevada					
North of 38° Latitude	10.5	9.5	9.5	9.5	9.5
South of 38° Latitude	9.5	9.5	9.5	9.5	9.5
New Hampshire	10.5	10.5	10.5	10.5	10.5
New Jersey	10.5	10.5	10.5	10.5	10.5
New Mexico					
North of 34° Latitude	9.5	9.0	9.0	9.5	9.5
South of 34° Latitude	9.5	9.0	9.0	9.0	9.5
New York	10.5	10.5	10.5	10.5	10.5
North Carolina	10.5	10.5	9.5	9.5	10.5
North Dakota	10.5	10.5	10.5	10.5	10.5
Ohio	10.5	10.5	10.5	10.5	10.5
Oklahoma	10.5	9.5	9.5	9.5	9.5
Oregon					
East of 122° Longitude	10.5	10.5	10.5	10.5	10.5
West of 122° Longitude	10.5	10.5	10.5	10.5	10.5
Pennsylvania	10.5	10.5	10.5	10.5	10.5
Rhode Island	10.5	10.5	10.5	10.5	10.5
South Carolina	10.5	10.5	9.5	9.5	10.5
South Dakota	10.5	10.5	10.5	10.5	10.5
Tennessee	10.5	10.5	9.5	9.5	10.5
Texas					
East of 99° Longitude	10.5	9.5	9.5	9.5	9.5
West of 99° Longitude	9.5	9.0	9.0	9.0	9.5
Utah	10.5	9.5	9.5	9.5	9.5
Vermont	10.5	10.5	10.5	10.5	10.5

¹Standards are expressed in pounds per square inch (psi).

APPLICABLE STANDARDS¹—Continued

State	May	June	July	Aug.	Sept.
Virginia.....	10.5	10.5	10.5	10.5	10.5
Washington:					
East of 122° Longitude.....	10.5	10.5	10.5	10.5	10.5
West of 122° Longitude.....	10.5	10.5	10.5	10.5	10.5
West Virginia.....	10.5	10.5	10.5	10.5	10.5
Wisconsin.....	10.5	10.5	10.5	10.5	10.5
Wyoming.....	10.5	10.5	10.5	10.5	10.5

¹ Standards are expressed in pounds per square inch (psi).² California areas include the following counties:

North Coast—Alameda, Contra Costa, Del Norte, Humboldt, Lake, Marin, Mendocino, Monterey, Napa, San Benito, San Francisco, San Mateo, Santa Clara, Santa Cruz, Solano, Sonoma, and Trinity.

Interior—Lesser, Modoc, Plumas, Sierra, Siskiyou, Alpine, Amador, Butte, Calaveras, Colusa, El Dorado, Fresno, Glenn, Kern (except that portion lying east of the Los Angeles County Aqueduct), Kings, Madera, Mariposa, Merced, Placer, Sacramento, San Joaquin, Shasta, Stanislaus, Sutter, Tehama, Tulare, Tuolumne, Yolo, Yuba, and Nevada.

South Coast—Orange, San Diego, San Luis Obispo, Santa Barbara, Ventura, and Los Angeles (except that portion north of the San Gabriel mountain range and east of the Los Angeles County Aqueduct).

Southeast—Imperial, Riverside, San Bernardino, Los Angeles (that portion north of the San Gabriel mountain range and east of the Los Angeles County Aqueduct), Mono, Inyo, and Kern (that portion lying east of the Los Angeles County Aqueduct).

(2) Applicable Standards¹ 1992 and Subsequent Years.

State	May	June	July	August	September
Alabama.....	9.0	7.8	7.8	7.8	7.8
Arizona.....	9.0	7.8	7.8	7.8	7.8
Arkansas.....	9.0	7.8	7.8	7.8	7.8
California.....	9.0	7.8	7.8	7.8	7.8
Colorado.....	9.0	7.8	7.8	7.8	7.8
Connecticut.....	9.0	9.0	9.0	9.0	9.0
Delaware.....	9.0	9.0	9.0	9.0	9.0
District of Columbia.....	9.0	7.8	7.8	7.8	7.8
Florida.....	9.0	7.8	7.8	7.8	7.8
Georgia.....	9.0	7.8	7.8	7.8	7.8
Idaho.....	9.0	9.0	9.0	9.0	9.0
Illinois.....	9.0	9.0	9.0	9.0	9.0
Indiana.....	9.0	9.0	9.0	9.0	9.0
Iowa.....	9.0	9.0	9.0	9.0	9.0
Kansas.....	9.0	7.8	7.8	7.8	7.8
Kentucky.....	9.0	9.0	9.0	9.0	9.0
Louisiana.....	9.0	7.8	7.8	7.8	7.8
Maine.....	9.0	9.0	9.0	9.0	9.0
Maryland.....	9.0	7.8	7.8	7.8	7.8
Massachusetts.....	9.0	9.0	9.0	9.0	9.0
Michigan.....	9.0	9.0	9.0	9.0	9.0
Minnesota.....	9.0	9.0	9.0	9.0	9.0
Mississippi.....	9.0	7.8	7.8	7.8	7.8
Missouri.....	9.0	7.8	7.8	7.8	7.8
Montana.....	9.0	9.0	9.0	9.0	9.0
Nebraska.....	9.0	9.0	9.0	9.0	9.0
Nevada.....	9.0	7.8	7.8	7.8	7.8
New Hampshire.....	9.0	9.0	9.0	9.0	9.0
New Jersey.....	9.0	9.0	9.0	9.0	9.0
New Mexico.....	9.0	7.8	7.8	7.8	7.8
New York.....	9.0	9.0	9.0	9.0	9.0
North Carolina.....	9.0	7.8	7.8	7.8	7.8
North Dakota.....	9.0	9.0	9.0	9.0	9.0
Ohio.....	9.0	9.0	9.0	9.0	9.0
Oklahoma.....	9.0	7.8	7.8	7.8	7.8
Oregon.....	9.0	7.8	7.8	7.8	7.8
Pennsylvania.....	9.0	9.0	9.0	9.0	9.0
Rhode Island.....	9.0	9.0	9.0	9.0	9.0
South Carolina.....	9.0	7.8	7.8	7.8	7.8
South Dakota.....	9.0	9.0	9.0	9.0	9.0

¹ Standards are expressed in pounds per square inch (psi).

State	May	June	July	August	September
Tennessee	9.0	7.8	7.8	7.8	7.8
Texas	9.0	7.8	7.8	7.8	7.8
Utah	9.0	7.8	7.8	7.8	7.8
Vermont	9.0	9.0	9.0	9.0	9.0
Virginia	9.0	7.8	7.8	7.8	7.8
Washington	9.0	9.0	9.0	9.0	9.0
West Virginia	9.0	9.0	9.0	9.0	9.0
Wisconsin	9.0	9.0	9.0	9.0	9.0
Wyoming	9.0	9.0	9.0	9.0	9.0

(b) *Determination of compliance.* Compliance with the standards listed in paragraph (a) of this section shall be determined by use of one of the sampling methodologies specified in Appendix D to this part and one of the testing methodologies specified in Appendix E to this part.

(c) *Liability.* Liability for violations of paragraph (a) of this section shall be determined according to the provisions of § 80.28.

(d) *Special provisions for alcohol blends.* (1) Any gasoline which meets the requirements of paragraph (d)(2) of this section and which is marketed in accordance with the requirements of paragraph (d)(3) of this section shall not be in violation of this section if its Reid vapor pressure does not exceed the applicable standard in paragraph (a) of this section by more than one pound per square inch (1.0 psi).

(2) In order to qualify for the special regulatory treatment specified in paragraph (d)(1) of this section, gasoline must contain at least 9% ethanol (by volume). The ethanol content of gasoline shall be determined by use of one of the testing methodologies specified in Appendix F to this part. The maximum ethanol content of gasoline shall not exceed any applicable waiver conditions under section 211(f)(4) of the Clean Air Act.

(3) In order to qualify for the special regulatory treatment specified in paragraph (d)(1) of this section, gasoline must be marketed in accordance with each of the following requirements:

(i) Each gasoline pump stand from which such gasoline is dispensed at a retail outlet or wholesale purchaser-consumer facility shall be affixed with a legible and conspicuous label which states that the gasoline dispensed from the pump contains ethanol and

the percentage concentration of ethanol.

(ii) Each invoice, loading ticket, bill of lading, delivery ticket and other document which accompanies the shipment of such gasoline shall contain a legible and conspicuous statement that the gasoline being shipped contains ethanol. Such documents shall be retained by distributors, resellers, carriers, ethanol blenders, retailers, and wholesale purchaser-consumers for at least one year, and shall be available for inspection by the Administrator or his authorized representative during such period.

(Approved by the Office of Management and Budget under control number 2060-0178)

[54 FR 11883, Mar. 22, 1989; 54 FR 27017, June 27, 1989, as amended at 54 FR 33219, Aug. 14, 1989; 55 FR 32666, June 11, 1990]

EFFECTIVE DATE NOTE: At 55 FR 23666, June 11, 1990, § 80.27 was amended by designating the table as paragraph (a)(1) and adding a paragraph heading and by adding a new paragraph (a)(2), effective July 11, 1990.

§ 80.28 Liability for violations of gasoline volatility controls and prohibitions.

(a) *Violations at refineries or importer facilities.* Where a violation of the applicable standard set forth in § 80.27 is detected at a refinery that is not an ethanol blending plant or at an importer's facility, the refiner or importer shall be deemed in violation.

(b) *Violations at carrier facilities.* Where a violation of the applicable standard set forth in § 80.27 is detected at a carrier's facility, whether in a transport vehicle, in a storage facility, or elsewhere at the facility, the following parties shall be deemed in violation:

(1) The carrier, except as provided in paragraph (g)(1) of this section; and

(2) The refiner (if he is not an ethanol blender) at whose refinery the gasoline was produced or the importer at whose import facility the gasoline was imported, except as provided in paragraph (g)(2) of this section; and

(3) The ethanol blender (if any) at whose ethanol blending plant the gasoline was produced, except as provided in paragraph (g)(6) of this section.

(c) *Violations at branded distributor facilities, reseller facilities, or ethanol blending plants.* Where a violation of the applicable standard set forth in § 80.27 is detected at a distributor facility, a reseller facility, or an ethanol blending plant which is operating under the corporate, trade, or brand name of a gasoline refiner or any of its marketing subsidiaries, the following parties shall be deemed in violation:

(1) The distributor or reseller, except as provided in paragraph (g)(3) of this section;

(2) The carrier (if any), if the carrier caused the gasoline to violate the applicable standard;

(3) The refiner under whose corporate, trade, or brand name (or that of any of its marketing subsidiaries) the distributor, reseller, or ethanol blender is operating, except as provided in paragraph (g)(4) of this section; and

(4) The ethanol blender (if any) at whose ethanol blending plant the gasoline was produced, except as provided in paragraph (g)(6) of this section.

(d) *Violations at unbranded distributor facilities or ethanol blending plants.* Where a violation of the applicable standard set forth in § 80.27 is detected at a distributor facility or an ethanol blending plant not operating under a refiner's corporate, trade, or brand name, or that of any of its marketing subsidiaries, the following parties shall be deemed in violation:

(1) The distributor, except as provided in paragraph (g)(3) of this section;

(2) The carrier (if any), if the carrier caused the gasoline to violate the applicable standard;

(3) The refiner (if he is not an ethanol blender) at whose refinery the gasoline was produced or the importer at whose import facility the gasoline was

imported, except as provided in paragraph (g)(2) of this section; and

(4) The ethanol blender (if any) at whose ethanol blending plant the gasoline was produced, except as provided in paragraph (g)(6) of this section.

(e) *Violations at branded retail outlets or wholesale purchaser-consumer facilities.* Where a violation of the applicable standard set forth in § 80.27 is detected at a retail outlet or at a wholesale purchaser-consumer facility displaying the corporate, trade, or brand name of a gasoline refiner or any of its marketing subsidiaries, the following parties shall be deemed in violation:

(1) The retailer or wholesale purchaser-consumer, except as provided in paragraph (g)(5) of this section;

(2) The distributor and/or reseller (if any), except as provided in paragraph (g)(3) of this section;

(3) The carrier (if any), if the carrier caused the gasoline to violate the applicable standard;

(4) The refiner whose corporate, trade, or brand name (or that of any of its marketing subsidiaries) is displayed at the retail outlet or wholesale purchaser-consumer facility, except as provided in paragraph (g)(4) of this section; and

(5) The ethanol blender (if any) at whose ethanol blending plant the gasoline was produced except as provided in paragraph (g)(6) of this section.

(f) *Violations at unbranded retail outlets or wholesale purchaser-consumer facilities.* Where a violation of the applicable standard set forth in § 80.27 is detected at a retail outlet or at a wholesale purchaser-consumer facility not displaying the corporate, trade, or brand name of a refiner or any of its marketing subsidiaries, the following parties shall be deemed in violation:

(1) The retailer or wholesale purchaser-consumer, except as provided in paragraph (g)(5) of this section;

(2) The distributor (if any), except as provided in paragraph (g)(3) of this section;

(3) The carrier (if any), if the carrier caused the gasoline to violate the applicable standard; and

(4) The ethanol blender (if any) at whose ethanol blending plant the gas-

oline was produced, except as provided in paragraph (g)(6) of this section.

(g) *Defenses.* (1) In any case in which a carrier would be in violation under paragraph (b)(1) of this section, the carrier shall not be deemed in violation if he can demonstrate:

(i) Bills of lading, invoices, delivery tickets, loading tickets or other documents from the refiner at whose refinery the gasoline was produced, the importer at whose facility the gasoline was imported, or the carrier, reseller, or distributor from whom the gasoline was received, which represented to the carrier that the gasoline was in compliance with the applicable standard when delivered to the carrier; and

(ii) Evidence of an oversight program conducted by the carrier, such as periodic sampling and testing of incoming gasoline, for monitoring the volatility of gasoline stored or transported by that carrier; and

(iii) That the violation was not caused by the carrier or his employee or agent.

(2) In any case in which a refiner or importer would be in violation under paragraph (b)(2) or (d)(3) of this section, the refiner or importer shall not be deemed in violation if he can demonstrate:

(i) That the violation was not caused by him or his employee or agent; and

(ii) Test results, performed in accordance with the sampling and testing methodologies set forth in Appendices D and E to this part, which evidence that the gasoline determined to be in violation was in compliance with the applicable standard when it was delivered to the next party in the distribution system.

(3) In any case in which a distributor or reseller would be in violation under paragraphs (c)(1), (d)(1), (e)(2), or (f)(2) of this section, the distributor or reseller shall not be deemed in violation if he can demonstrate:

(i) That the violation was not caused by him or his employee or agent; and

(ii) Bills of lading, invoices, delivery tickets, loading tickets or other documents from the refiner at whose refinery the gasoline was produced, the importer at whose facility the gasoline was imported, or the carrier, reseller or distributor from whom the gasoline

was received, which represented to the distributor or reseller that the gasoline was in compliance with the applicable standard when delivered to the distributor or reseller; and

(iii) Evidence of an oversight program conducted by the distributor or reseller, such as periodic sampling and testing of gasoline, for monitoring the volatility of gasoline that the distributor or reseller sells, supplies, offers for sale or supply, or transports.

(4) In any case in which a refiner would be in violation under paragraphs (c)(3) or (e)(4) of this section, the refiner shall not be deemed in violation if he can demonstrate all of the following:

(i) Test results, performed in accordance with the sampling and testing methodologies set forth in Appendices D and E to this part at the refinery at which the gasoline was produced, which evidence that the gasoline determined to be in violation was in compliance with the applicable standard when transported from the refinery; and

(ii) That the violation was not caused by him or his employee or agent; and

(iii) That the violation:

(A) Was caused by an act in violation of law (other than the Act or this part), or an act of sabotage or vandalism, whether or not such acts are violations of law in the jurisdiction where the violation of the requirements of this part occurred, or

(B) Was caused by the action of a reseller, an ethanol blender, or a retailer supplied by such reseller or ethanol blender, in violation of a contractual undertaking imposed by the refiner on such reseller or ethanol blender designed to prevent such action, and despite reasonable efforts by the refiner (such as periodic sampling and testing) to insure compliance with such contractual obligation, or

(C) Was caused by the action of a retailer who is supplied directly by the refiner (and not by a reseller), in violation of a contractual undertaking imposed by the refiner on such retailer designed to prevent such action, and despite reasonable efforts by the refiner (such as periodic sampling and test-

ing) to insure compliance with such contractual obligation, or

(D) Was caused by the action of a distributor or an ethanol blender subject to a contract with the refiner for transportation of gasoline from a terminal to a distributor, ethanol blender, retailer or wholesale purchaser-consumer, in violation of a contractual undertaking imposed by the refiner on such distributor or ethanol blender designed to prevent such action, and despite reasonable efforts by the refiner (such as periodic sampling and testing) to insure compliance with such contractual obligation, or

(E) Was caused by a carrier or other distributor not subject to a contract with the refiner but engaged by him for transportation of gasoline from a terminal to a distributor, ethanol blender, retailer or wholesale purchaser-consumer, despite reasonable efforts by the refiner (such as specification or inspection of equipment) to prevent such action, or

(F) Occurred at a wholesale purchaser-consumer facility: *Provided, however*, That if such wholesale purchaser-consumer was supplied by a reseller or ethanol blender, the refiner must demonstrate that the violation could not have been prevented by such reseller's or ethanol blender's compliance with a contractual undertaking imposed by the refiner on such reseller or ethanol blender as provided in paragraph (g)(4)(iii)(B) of this section.

(iv) In paragraphs (g)(4)(iii)(A) through (E) of this section, the term "was caused" means that the refiner must demonstrate by reasonably specific showings, by direct or circumstantial evidence, that the violation was caused or must have been caused by another.

(5) In any case in which a retailer or wholesale purchaser-consumer would be in violation under paragraphs (e)(1) or (f)(1) of this section, the retailer or wholesale purchaser-consumer shall not be deemed in violation if he can demonstrate that the violation was not caused by him or his employee or agent.

(6) In any case in which an ethanol blender would be in violation under paragraphs (b)(3), (c)(4), (d)(4), (e)(5) or (f)(4) of this section, the ethanol

blender shall not be deemed in violation if he can demonstrate:

(i) That the violation was not caused by him or his employee or agent; and

(ii) Bills of lading, invoices, delivery tickets, loading tickets or other documents from the refiner at whose refinery the gasoline was produced, the importer at whose facility the gasoline was imported, or the carrier, reseller, or distributor from whom the gasoline was received, which represented to the ethanol blender that the gasoline to which ethanol was added was in compliance with the applicable standard when delivered to the ethanol blender; and

(iii) Evidence of an oversight program conducted by the ethanol blender, such as periodic sampling and testing of gasoline, for monitoring the volatility of gasoline that the ethanol blender sells, supplies, offers for sale or supply, or transports; and

(iv) That the gasoline determined to be in violation contained no more than 10% ethanol (by volume) when it was delivered to the next party in the distribution system.

(7) In paragraphs (g)(1)(iii), (g)(2)(i), (g)(3)(i), (g)(4)(ii), (g)(5), and (g)(6)(i) of this section the respective party must demonstrate by reasonably specific showings, by direct or circumstantial evidence, that it or its employee or agent did not cause the violation.

[54 FR 11885, Mar. 22, 1989; 54 FR 27017, June 27, 1989]

APPENDIX A—TEST FOR THE DETERMINATION OF PHOSPHORUS IN GASOLINE

1. Scope.

1.1 This method was developed for the determination of phosphorus generally present as pentavalent phosphate esters or salts, or both, in gasoline. This method is applicable for the determination of phosphorus in the range from 0.0008 to 0.15 g P/U.S. gal, or 0.2 to 49 mg P/liter.

2. Applicable documents.

2.1 ASTM Standards:

D 1100 Specification for Filter Paper for Use in Chemical Analysis.

3. Summary of method.

3.1 Organic matter in the sample is decomposed by ignition in the presence of zinc oxide. The residue is dissolved in sulfuric acid and reacted with ammonium molybdate

and hydrazine sulfate. The absorbance of the "Molybdenum Blue" complex is proportional to the phosphorus concentration in the sample and is read at approximately 820 nm in a 5-cm cell.

4. Apparatus.

4.1 Buret, 10-ml capacity, 0.05-ml subdivisions.

4.2 Constant-Temperature Bath, equipped to hold several 100-ml volumetric flasks submerged to the mark. Bath must have a large enough reservoir or heat capacity to keep the temperature at 180 to 190° F (82.2 to 87.8° C) during the entire period of sample heating.

NOTE 1: If the temperature of the hot water bath drops below 180° F (82.2° C) the color development may not be complete.

4.3 Cooling Bath, equipped to hold several 100-ml volumetric flasks submerged to the mark in ice water.

4.4 Filter Paper, for quantitative analysis, Class G for fine precipitates as defined in Specification D 1100.

4.5 Ignition Dish—Coors porcelain evaporating dish, glazed inside and outside, with pourout (size no. 00A, diameter 75 mm, capacity 70 ml).

4.6 Spectrophotometer, equipped with a tungsten lamp, a red-sensitive phototube capable of operating at 830 nm and with absorption cells that have a 5-cm light path.

4.7 Thermometer, range 50 to 220° F (10 to 105° C).

4.8 Volumetric Flask, 100-ml with ground-glass stopper.

4.9 Volumetric Flask, 1000-ml with ground-glass stopper.

4.10 Syringe, Luer-Lok, 10-ml equipped with 5-cm, 22-gage needle.

5. Reagents.

5.1 Purity of Reagents—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 Purity of Water—Unless otherwise indicated, references to water shall be understood to mean distilled water or water of equal purity.

5.3 Ammonium Molybdate Solution—Using graduated cylinders for measurement add slowly (Note 2), with continuous stirring, 225 ml of concentrated sulfuric acid to 500 ml of water contained in a beaker placed in a bath of cold water. Cool to room temperature and add 20 g of ammonium molybdate tetrahydrate ($(\text{NH}_4)_6\text{Mo}_7\text{O}_{24} \cdot 4\text{H}_2\text{O}$). Stir until solution is complete and transfer to a

1000-ml flask. Dilute to the mark with water.

NOTE 2: Wear a face shield, rubber gloves, and a rubber apron when adding concentrated sulfuric acid to water.

5.4 Hydrazine Sulfate Solution—Dissolve 1.5 of hydrazine sulfate ($\text{H}_2\text{NNH}_2 \cdot \text{H}_2\text{SO}_4$) in 1 litre of water, measured with a graduated cylinder.

NOTE 3: This solution is not stable. Keep it tightly stoppered and in the dark. Prepare a fresh solution after 3 weeks.

5.5 Molybdate-Hydrazine Reagent—Pipet 25 ml of ammonium molybdate solution into a 100-ml volumetric flask containing approximately 50 ml of water, add by pipet 10 ml of $\text{N}_2\text{NNH}_2 \cdot \text{H}_2\text{SO}_4$ solution, and dilute to 100 ml with water.

NOTE 4: This reagent is unstable and should be used within about 4 h. Prepare it immediately before use. Each determination (including the blank) uses 50 ml.

5.6 Phosphorus, Standard Solution (10.0 $\mu\text{g P/ml}$)—Pipet 10 ml of stock standard phosphorus solution into a 1000-ml volumetric flask and dilute to the mark with water.

5.7 Phosphorus, Stock Standard Solution (1.00 mg P/ml)—Dry approximately 5 g of potassium dihydrogen phosphate (KH_2PO_4) in an oven at 221 to 230° F (105 to 110° C) for 3 h. Dissolve 4.393 ± 0.002 g of the reagent in 150 ml, measured with a graduated cylinder, of H_2SO_4 (1+10) contained in a 1000-ml volumetric flask. Dilute with water to the mark.

5.8 Sulfuric Acid (1+10)—Using graduated cylinders for measurement add slowly (Note 2), with continuous stirring, 100-ml of concentrated sulfuric acid (H_2SO_4 , sp gr 1.84) to 1 litre of water contained in a beaker placed in a bath of cold water.

5.9 Zinc Oxide.

NOTE 5: High-bulk density zinc oxide may cause spattering. Density of approximately 0.5 g/cm³ has been found satisfactory.

6. Calibration.

6.1 Transfer by buret, or a volumetric transfer pipet, 0.0, 0.5, 1.0, 1.5, 2.0, 3.0, 3.5, and 4.0 ml of phosphorus standard solution into 100-ml volumetric flasks.

6.2 Pipet 10 ml of H_2SO_4 (1+10) into each flask. Mix immediately by swirling.

6.3 Prepare the molybdate-hydrazine solution. Prepare sufficient volume of reagent based on the number of samples being analyzed.

6.4 Pipet 50 ml of the molybdate-hydrazine solution to each volumetric flask. Mix immediately by swirling.

6.5 Dilute to 100 ml with water.

6.6 Mix well and place in the constant-temperature bath so that the contents of the flask are submerged below the level of the bath. Maintain bath temperature at 180 to 190° F (82.2 to 87.8° C) for 25 min (Note 1).

6.7 Transfer the flask to the cooling bath and cool the contents rapidly to room temperature. Do not allow the samples to cool more than 5° F (2.8° C) below room temperature.

NOTE 6: Place a chemically clean thermometer in one of the flasks to check the temperature.

6.8 After cooling the flasks to room temperature, remove them from the cooling water bath and allow them to stand for 10 min. at room temperature.

6.9 Using the 2.0-ml phosphorus standard in a 5-cm cell, determine the wavelength near 820 nm that gives maximum absorbance. The wavelength giving maximum absorbance should not exceed 830 nm.

6.9.1 Using a red-sensitive phototube and 5-cm cells, adjust the spectrophotometer to zero absorbance at the wavelength of maximum absorbance using distilled water in both cells. Use the wavelength of maximum absorbance in the determination of calibration readings and future sample readings.

6.9.2 The use of 1-cm cells for the higher concentrations is permissible.

6.10 Measure the absorbance of each calibration sample including the blank (0.0 ml phosphorus standard) at the wavelength of maximum absorbance with distilled water in the reference cell.

NOTE 7: Great care must be taken to avoid possible contamination. If the absorbance of the blank exceeds 0.04 (for 5-cm cell), check for source of contamination. It is suggested that the results be disregarded and the test be rerun with fresh reagents and clean glassware.

6.11 Correct the absorbance of each standard solution by subtracting the absorbance of the blank (0 ml phosphorus standard).

6.12 Prepare a calibration curve by plotting the corrected absorbance of each standard solution against micrograms of phosphorus. One millilitre of phosphorus standard solution provides 10 µg of phosphorus.

7. Sampling.

7.1 Selection of the size of the sample to be tested depends on the expected concentration of phosphorus in the sample. If a concentration of phosphorus is suspected to be less than 0.0038 g/gal (1.0 mg/litre), it will be necessary to use 10 ml of sample.

NOTE 8: Two grams of zinc oxide cannot absorb this volume of gasoline. Therefore the 10-ml sample is ignited in aliquots of 2 ml in the presence of 2 g of zinc oxide.

7.2 The following table serves as a guide for selecting sample size:

Phosphorus, milligrams per liter	Equivalent, grams per gallon	Sample size, milliliter
2.5 to 40	0.01 to 0.15	1.00
1.3 to 20	0.005 to 0.075	2.00

Phosphorus, milligrams per liter	Equivalent, grams per gallon	Sample size, milliliter
0.9 to 13	0.0037 to 0.05	3.00
1 or less	0.0038 or less	10.00

8. Procedure.

8.1 Transfer 2±0.2 g of zinc oxide into a conical pile in a clean, dry, unetched ignition dish.

NOTE 9: In order to obtain satisfactory accuracy with the small amounts of phosphorus involved, it is necessary to take extensive precautions in handling. The usual precautions of cleanliness, careful manipulation, and avoidance of contamination should be scrupulously observed; also, all glassware should be cleaned before use, with cleaning acid or by some procedure that does not involve use of commercial detergents. These compounds often contain alkali phosphates which are strongly adsorbed by glass surfaces and are not removed by ordinary rinsing. It is desirable to segregate a special stock of glassware for use only in the determination of phosphorus.

8.2 Make a deep depression in the center of the zinc oxide pile with a stirring rod.

8.3 Pipet the gasoline sample (Note 10) (see 7.2 for suggested sample volume) into the depression in the zinc oxide. Record the temperature of the fuel if the phosphorus content is required at 60° F (15.6° C) and make correction as directed in 9.2.

NOTE 10: For the 10-ml sample use multiple additions and a syringe. Hold the tip of the needle at approximately ¾ of the depth of the zinc oxide layer and slowly deliver 2 ml of the sample; fast sample delivery may give low results. Give sufficient time for the gasoline to be absorbed by the zinc oxide. Follow step 8.6. Cool the dish to room temperature. Repeat steps 8.3 and 8.6 until all the sample has been burned. Safety—cool the ignition dish before adding the additional aliquots of gasoline to avoid a flash fire.

8.4 Cover the sample with a small amount of fresh zinc oxide from reagent bottle (use the tip of a small spatula to deliver approximately 0.2 g). Tap the sides of the ignition dish to pack the zinc oxide.

8.5 Prepare the blank, using the same amount of zinc oxide in an ignition dish.

8.6 Ignite the gasoline, using the flame from a bunsen burner. Allow the gasoline to burn to extinction (NOTE 10).

8.7 Place the ignition dishes containing the sample and blank in a hot muffle furnace set at a temperature of 1150 to 1300° F (621 to 704° C) for 10 min. Remove and cool the ignition dishes. When cool gently tap the sides of the dish to loosen the zinc oxide. Again place the dishes in the muffle furnace for 5 min. Remove and cool the dishes.

tion dishes to room temperature. The above treatment is usually sufficient to burn the carbon. If the carbon is not completely burned off place the dish into the oven for further 5-min. periods.

Note 11: Step 8.7 may also be accomplished by heating the ignition dish with a Meker burner gradually increasing the intensity of heat until the carbon from the sides of the dish has been burned, then cool to room temperature.

8.8 Pipet 25 ml of H₂SO₄ (1+10) to each ignition dish. While pipeting, carefully wash all traces of zinc oxide from the sides of the ignition dish.

8.9 Cover the ignition dish with a borosilicate watch glass and warm the ignition dish on a hot plate until the zinc oxide is completely dissolved.

8.10 Transfer the solution through filter paper to a 100-ml volumetric flask. Rinse the watch glass and the dish several times with distilled water (do not exceed 25 ml) and transfer the washings through the filter paper to the volumetric flask.

8.11 Prepare the molybdate-hydrazine solution.

8.12 Add 50 ml of the molybdate-hydrazine solution by pipet to each 100-ml volumetric flask. Mix immediately by swirling.

8.13 Dilute to 100 ml with water and mix well. Remove stoppers from flasks after mixing.

8.14 Place the 100-ml flasks in the constant-temperature bath for 25 min. so that the contents of the flasks are below the liquid level of the bath. The temperature of the bath should be 180 to 190° F (82.2 to 87.8° C) (NOTE 1).

8.15 Transfer the 100-ml flasks to the cooling bath and cool the contents rapidly to room temperature (NOTE 6).

8.16 Allow the samples to stand at room temperature before measuring the absorbance.

Note 12: The color developed is stable for at least 4 h.

8.17 Set the spectrophotometer to the wavelength of maximum absorbance as determined in 6.9. Adjust the spectrophotometer to zero absorbance, using distilled water in both cells.

8.18 Measure the absorbance of the samples at the wavelength of maximum absorbance with distilled water in the reference cell.

8.19 Subtract the absorbance of the blank from the absorbance of each sample (NOTE 7).

8.20 Determine the micrograms of phosphorous in the sample, using the calibration curve from 6.12 and the corrected absorbance.

Calculations.

8.1 Calculate the milligrams of phosphorous per litre of sample as follows:

$P, \text{ mg/litre} = P/V$

where:

P=micrograms of phosphorus read from calibration curve, and

V=millilitres of gasoline sample.

To convert to grams of phosphorus per U.S. gallon of sample, multiply mg P/litre by 0.0038.

9.2 If the gasoline sample was taken at a temperature other than 60° F (15.6° C) make the following temperature correction:

$\text{mg P/litre at } 15.6^{\circ} \text{ C} = [\text{mg P/litre at } t] [1 + 0.001 (t - 15.6)]$

where:

t=observed temperature of the gasoline, ° C.

9.3 Concentrations below 2.5 mg/litre or 0.01 g/gal should be reported to the nearest 0.01 mg/litre or 0.0001 g/U.S. gal.

9.3.1 For higher concentrations, report results to the nearest 1 mg P/litre or 0.005 g P/U.S. gal.

10. Precision.

10.1 The following criteria should be used for judging the acceptability of results (95 percent confidence):

10.2 Repeatability—Duplicate results by the same operator should be considered suspect if they differ by more than the following amounts:

g P/U.S. gal (mg P/litre)	Repeatability
0.0008 to 0.005 (0.2 to 1.3).....	0.0002 g P/U.S. gal (0.05 mg P/litre)
0.005 to 0.15 (1.3 to 40).....	7% of the mean. //

10.3 Reproducibility—The results submitted by each of two laboratories should not be considered suspect unless they differ by more than the following amounts:

g P/U.S. gal (mg P/litre)	Reproducibility
0.0008 to 0.005 (0.2 to 1.3).....	0.0005 g P/U.S. gal (0.13 mg P/litre).
0.005 to 0.15 (1.3 to 40).....	13% of the mean.

[39 FR 24891, July 8, 1974; 39 FR 25653, July 12, 1974]

APPENDIX B—TEST METHODS FOR LEAD IN GASOLINE

METHOD 1—STANDARD METHOD TEST FOR LEAD IN GASOLINE BY ATOMIC ABSORPTION SPECTROMETRY

1. Scope.

1.1. This method covers the determination of the total lead content of gasoline. The

procedure's calibration range is 0.010 to 0.10 gram of lead/U.S. gal. Samples above this level should be diluted to fall within this range or a higher level calibration standard curve must be prepared. The higher level curve must be shown to be linear and measurement of lead at these levels must be shown to be accurate by the analysis of control samples at a higher level of alkyl lead content. The method compensates for variations in gasoline composition and is independent of lead alkyl type.

2. Summary of method.

2.1 The gasoline sample is diluted with methyl isobutyl ketone and the alkyl lead compounds are stabilized by reaction with iodine and a quarternary ammonium salt. The lead content of the sample is determined by atomic absorption flame spectrometry at 2833 Å, using standards prepared from reagent grade lead chloride. By the use of this treatment, all alkyl lead compounds give identical response.

3. Apparatus.

3.1 Atomic Absorption Spectrometer, capable of scale expansion and nebulizer adjustment, and equipped with a slot burner and premix chamber for use with an air-acetylene flame.

3.2 Volumetric Flasks, 50-ml, 100-ml, 250-ml, and one litre sizes.

3.3 Pipets, 2-ml, 5-ml, 10-ml, 20-ml, and 50-ml sizes.

3.4 Micropipet, 100- μ l, Eppendorf type or equivalent.

4. Reagents.

4.1 Purity of Reagents—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

4.2 Purity of Water—Unless otherwise indicated, references to water shall be understood to mean distilled water or water of equal purity.

4.3 Aliquat 336 (tricapryl methyl ammonium chloride).

4.4 Aliquat 336/MIBK Solution (10 percent v/v)—Dissolve and dilute 100 ml (88.0 g) of Aliquat 336 with MIBK to one liter.

4.5 Aliquat 336/MIBK Solution (1 percent v/v)—Dissolve and dilute 10 ml (8.8 g) of Aliquat 336 with MIBK to one liter.

4.6 Iodine Solution—Dissolve and dilute 3.0 g iodine crystals with Toluene to 100 ml.

4.7 Lead Chloride.

4.8 Lead-Sterile Gasoline—Gasoline containing less than 0.005 g Pb/gal.

4.9 Lead, Standard Solution (5.0 g Pb/gal)—Dissolve 0.4433 g of lead chloride (PbCl₂) previously dried at 105° C for 3 h in about 200 ml of 10 percent Aliquat 336/MIBK solution in a 250-ml volumetric flask. Dilute to the mark with the 10 percent Aliquat solution, mix, and store in a brown bottle having a polyethylene-lined cap. This solution contains 1,321 μ g Pb/ml, which is equivalent to 5.0 g Pb/gal.

4.10 Lead, Standard Solution (1.0 g Pb/gal)—By means of a pipet, accurately transfer 50.0 ml of the 5.0 g Pb/gal solution to a 250-ml volumetric flask, dilute to volume with 1 percent Aliquat/MIBK solution. Store in a brown bottle having a polyethylene-lined cap.

4.11 Lead, Standard Solutions (0.02, 0.05, and 0.10 g Pb/gal)—Transfer accurately by means of pipets 2.0, 5.0, and 10.0 ml of the 1.0-g Pb/gal solution to 100-ml volumetric flasks; add 5.0 ml of 1 percent Aliquat 336 solution to each flask; dilute to the mark with MIBK. Mix well and store in bottles having polyethylene-lined caps.

4.12 Methyl Isobutyl Ketone (MIBK). (4-methyl-2-pentanone).

5. Calibration.

5.1 Preparation of Working Standards—Prepare three working standards and a blank using the 0.02, 0.05, and 0.10-g Pb/gal standard lead solutions described in 4.11.

5.1.1 To each of four 50-ml volumetric flasks containing 30 ml of MIBK, add 5.0 ml of low lead standard solution and 5.0 ml of lead-free gasoline. In the case of the blank, add only 5.0 ml of lead-free gasoline.

5.1.2 Add immediately 0.1 ml of iodine/toluene solution by means of the 100- μ l Eppendorf pipet. Mix well.¹

5.1.3 Add 5 ml of 1 percent Aliquat 336 solution and mix.

5.1.4 Dilute to volume with MIBK and mix well.

5.2 Preparation of Instrument—Optimize the atomic absorption equipment for lead at 2833 Å. Using the reagent blank, adjust the gas mixture and the sample aspiration rate to obtain an oxidizing flame.

5.2.1 Aspirate the 0.1-g Pb/gal working standard and adjust the burner position to give maximum response. Some instruments require the use of scale expansion to produce a reading of 0.150 to 0.170 for this standard.

5.2.2 Aspirate the reagent blank to zero the instrument and check the absorbances of the three working standards for linearity.

6. Procedure.

¹EPA practice will be to mix well by shaking vigorously for approximately one minute.

6.1 To a 50 ml volumetric flask containing 30 ml MIBK, add 5.0 ml of gasoline sample and mix.²

6.1.1 Add 0.10 ml (100 μ l) of iodine/toluene solution and allow the mixture to react about 1 min.³

6.1.2 Add 5.0 ml of 1 percent Aliquot 336/MIBK solution and mix.

6.1.3 Dilute to volume with MIBK and mix.

6.2 Aspirate the samples and working standards and record the absorbance values with frequent checks of the zero.

6.3 Any sample resulting in a peak greater than 0.05 g Pb/gal will be run in duplicate. Samples registering greater than 0.10 g Pb/gal should be diluted with iso-octane or unleaded fuel to fall within the calibration range or a higher level calibration standard curve must be prepared. The higher level curve must be shown to be linear and measurement of lead at these levels must be shown to be accurate by the analysis of control samples at a higher level of alkyl lead content.

7. Calculations.

7.1 Plot the absorbance values versus concentration represented by the working standards and read the concentrations of the samples from the graph.

8. Precision.

8.1 The following criteria should be used for judging the acceptability of results (95 percent confidence):

8.1.1 Repeatability—Duplicate results by the same operator should be considered suspect if they differ by more than 0.005 g/gal.

8.1.2 Reproducibility—The results submitted by each of two laboratories should not be considered suspect unless the two results differ by more than 0.01 g/gal.

METHOD 2—AUTOMATED METHOD TEST FOR LEAD IN GASOLINE BY ATOMIC ABSORPTION SPECTROMETRY

1. Scope and application.

1.1 This method covers the determination of the total lead content of gasoline. The procedure's calibration range is 0.010 to 0.10 gram of lead/U.S. gal. Samples above this level should be diluted to fall within this range or a higher level calibration standard curve must be prepared. The higher level curve must be shown to be linear and measurement of lead at these levels must be shown to be accurate by the analysis of control samples at a higher level of alkyl lead content. The method compensates for variations in gasoline composition and is independent of lead alkyl type.

²The gasoline should be allowed to come to room temperature (25° C).

³See footnote at end of Appendix C.

1.2 This method may be used as an alternative to the Standard Method set forth above.

1.3 Where trade names or specific products are noted in the method, equivalent apparatus and chemical reagents may be used. Mention of trade names or specific products is for the assistance of the user and does not constitute endorsement by the U.S. Environmental Protection Agency.

2. Summary of method.

2.1 The gasoline sample is diluted with methyl isobutyl ketone (MIBK) and the alkyl lead compounds are stabilized by reacting with iodine and a quarternary ammonium salt. An automated system is used to perform the diluting and the chemical reactions and feed the products to the atomic absorption spectrometer with an air-acetylene flame.

2.2 The dilution of the gasoline with MIBK compensates for severe non-atomic absorption, scatter from unburned carbon containing species and matrix effects caused in part by the burning characteristics of gasoline.

2.3 The *in-situ* reaction of alkyl lead in gasoline with iodine eliminates the problem of variations in response due to different alkyl types by leveling the response of all alkyl lead compounds.

2.4 The addition of the quarternary ammonium salt improves response and increases the stability of the alkyl iodide complex.

3. Sample handling and preservation.

3.1 Samples should be collected and stored in containers which will protect them from changes in the lead content of the gasoline such as from loss of volatile fractions of the gasoline by evaporation or leaching of the lead into the container or cap.

3.2 If samples have been refrigerated they should be brought to room temperature prior to analysis.

4. Apparatus.

4.1 AutoAnalyzer system consisting of:

4.1.1 Sampler 20/hr cam, 30/hr cam.

4.1.2 Proportioning pump.

4.1.3 Lead in gas manifold.

4.1.4 Disposable test tubes.

4.1.5 Two 2-liter and one 0.5 liter Erlenmeyer solvent displacement flasks. Alternatively, high pressure liquid chromatography (HPLC) or syringe pumps may be used.

4.2 Atomic Absorption Spectroscopy (AAS) Detector System consisting of:

4.2.1 Atomic absorption spectrometer.

4.2.2 10" strip chart recorder.

4.2.3 Lead hollow cathode lamp or electrodeless discharge lamp (EDL).

5. Reagents.

5.1 Aliquot 336/MIBK solution (10% v/v): Dissolve and dilute 100 ml (88.0 g) of Ali-

quat 336 (Aldrich Chemical Co., Milwaukee, Wisconsin) with MIBK (Burdick & Jackson Lab., Inc., Muskegon, Michigan) to one liter.

5.2 Aliquat 336/iso-octane solution (1% v/v): Dissolve and dilute 10 ml (8.8 g) of Aliquat 336 (reagent 5.1) with iso-octane to one liter.

5.3 Iodine solution (3% w/v): Dissolve and dilute 3.0 g iodine crystals (American Chemical Society) with toluene (Burdick & Jackson Lab., Inc., Muskegon, Michigan) to 100 ml.

5.4 Iodine working solution (0.24% w/v): Dilute 8 ml of reagent 5.3 to 100 ml with toluene.

5.5 Methyl isobutyl ketone (MIBK) (4-methyl-2-pentanone).

5.6 Certified unleaded gasoline (Phillips Chemical Co., Borger, Texas) or iso-octane (Burdick & Jackson Lab., Inc., Muskegon, Michigan).

6. Calibration standards.

6.1 Stock 5.0 g Pb/gal Standard:

Dissolve 0.4433 gram of lead chloride ($PbCl_2$) previously dried at 105° C for 3 hours in 200 ml of 10% v/v Aliquat 336/MIBK solution (reagent 5.1) in a 250 ml volumetric flask. Dilute to volume with reagent 5.1 and store in an amber bottle.

6.2 Intermediate 1.0 g Pb/gal Standard:

Pipet 50 ml of the 5.0 g Pb/gal standard into a 250 ml volumetric flask and dilute to volume with a 1% v/v Aliquat 336/iso-octane solution (reagent 5.2). Store in an amber bottle.

6.3 Working 0.02, 0.05, 0.10 g Pb/gal Standards:

Pipet 2.0, 5.0, and 10.0 ml of the 1.0 g Pb/gal solution to 100 ml volumetric flasks. Add 5 ml of a 1% Aliquat 336/iso-octane solution to each flask. Dilute to volume with iso-octane. These solutions contain 0.02, 0.05, and 0.10 g Pb/gal in a 0.05% Aliquat 336/iso-octane solution.

7. AAS Instrumental conditions.

7.1 Lead hollow cathode lamp.

7.2 Wavelength: 283.3 nm.

7.3 Slit: 4 (0.7mm).

7.4 Range: UV.

7.5 Fuel: Acetylene (approx. 20 ml/min at 8 psi).

7.6 Oxidant: Air (approx. 65 ml/min at 31 psi).

7.7 Nebulizer: 5.2 ml/min.

7.8 Chart speed: 10 in/hr.

8. Procedures.

8.1 AAS start-up.

8.1.1 Assure that instrumental conditions have been optimized and aligned according to Section 7 and the instrument has had substantial time for warm-up.

8.2 Auto Analyzer start-up [see figure 1].

8.2.1 Check all pump tubing and replace as necessary. Iodine tubing should be changed daily. All pump tubing should be

replaced after one week of use. Place the platen on the pump.

8.2.2 Withdraw any water from the sample wash cup and fill with certified unleaded gasoline (reagent 5.6).

8.2.3 Fill the 2-liter MIBK dilution displacement Erlenmeyer flask (reagent 5.5) and the 0.5 liter Aliquat 336/MIBK 1% v/v (reagent 5.2) displacement flask and place the rubber stopper glass tubing assemblies in their respective flasks.

8.2.4 Fill a 2-liter Erlenmeyer flask with distilled water. The water will be used to displace the solvents. Therefore, place the appropriate lines in this flask. This procedure is not relevant if syringe pumps are used.

8.2.5 Fill the final debubbler reverse displacement 2-liter Erlenmeyer flask with distilled water and place the rubber stopper glass tubing assembly in the flask.

8.2.6 Place the appropriate lines for the iodine reagent (reagent 5.4) and the wash solution (reagent 5.6) in their respective bottles.

8.2.7 Start the pump and connect the aspiration line from the manifold to the AAS.

8.2.8 Some initial checks to assure that the reagents are being added are:

a. A good uniform bubble pattern.

b. Yellow color evident due to iodine in the system.

c. No surging in any tubing.

8.3 Calibration.

8.3.1 Turn the chart drive on and obtain a steady baseline.

8.3.2 Load standards and samples into sample tray.

8.3.3 Start the sampler and run the standards (Note: first check the sample probe positioning with an empty test tube).

8.3.4 Check the linearity of calibration standards response and slope by running a least squares fit. Check these results against previously obtained results. They should agree within 10%.

8.3.5 If the above is in control then start the sample analysis.

8.4 Sample Analysis.

8.4.1 To minimize gasoline vapor in the laboratory, load the sample tray about 5-10 test tubes ahead of the sampler.

8.4.2 Record the sample number on the strip chart corresponding to the appropriate peak.

8.4.3 Every ten samples run the high calibration standard and a previously analyzed sample (duplicate). Also let the sampler skip to check the baseline.

8.4.4 After an acceptable peak (within the calibration range) is obtained, pour the excess sample from the test tube into the waste gasoline can.

8.4.5 Any sample resulting in a peak greater than 0.05 g Pb/gal will be run in duplicate. Samples registering greater than

0.10 g Pb/gal should be diluted with iso-octane or unleaded fuel to fall within the calibration range or a higher level calibration standard curve must be prepared. The higher level curve must be shown to be linear and measurement of lead at these levels must be shown to be accurate by the analysis of control samples at a higher level of alkyl lead content.

8.5 Shut Down.

8.5.1 Replace the solvent displacement flask with flasks filled with distilled water. Also place all other lines in a beaker of distilled water. Rinse the system with distilled water for 15 minutes.

8.5.2 Withdraw the gasoline from the wash cup and fill with water.

8.5.3 Dispose of all solvent waste in waste glass bottles.

8.5.4 Turn the AAS off after extinguishing the flame. Also turn the recorder and pump off. Remove the platen and release the pump tubing.

8.5.5 shut the acetylene off at the tank and bleed the line.

9. Quality control.

9.1 Precision.

9.1.1 All duplicate results should be considered suspect if they differ by more than 0.005 g Pb/gal.

9.2 Accuracy.

9.2.1 All quality control standard checks should agree within 10% of the nominal value of the standard.

9.2.2 All spikes should agree within 10% of the known addition.

10. Past quality control data.

10.1 Precision.

10.1.1 Duplicate analysis for 156 samples in a single laboratory has resulted in an average difference of 0.00011 g Pb/gal with a standard deviation of 0.0023.

10.1.2 Replicate analysis in a single laboratory (greater than 5 determinations) of samples at concentrations of 0.010, 0.048, and 0.085 g Pb/gal resulted in relative standard deviations of 4.2%, 3.5%, and 3.3% respectively.

10.2 Accuracy.

10.2.1 The analysis of National Bureau of Standards (NBS) lead in reference fuel of known concentrations in a single laboratory has resulted in found values deviating from the true value for 11 determinations of 0.0322 g Pb/gal by an average of 0.56% with a standard deviation of 6.8%, for 15 determinations of 0.0519 g Pb/gal by an average of -1.1% with a standard deviation of 5.8%, and for 7 determinations of 0.0725 g Pb/gal by an average of 3.5% with a standard deviation of 4.8%.

10.2.2 Twenty-three analyses of blind reference samples in a single laboratory (U.S. EPA, RTP, N.C.) have resulted in found values differing from the true value by an average of -0.0009 g Pb/gal with a standard deviation of 0.004.

10.2.3 In a single laboratory, the average percent recovery of 108 spikes made to samples was 101% with a standard deviation of 5.6%.

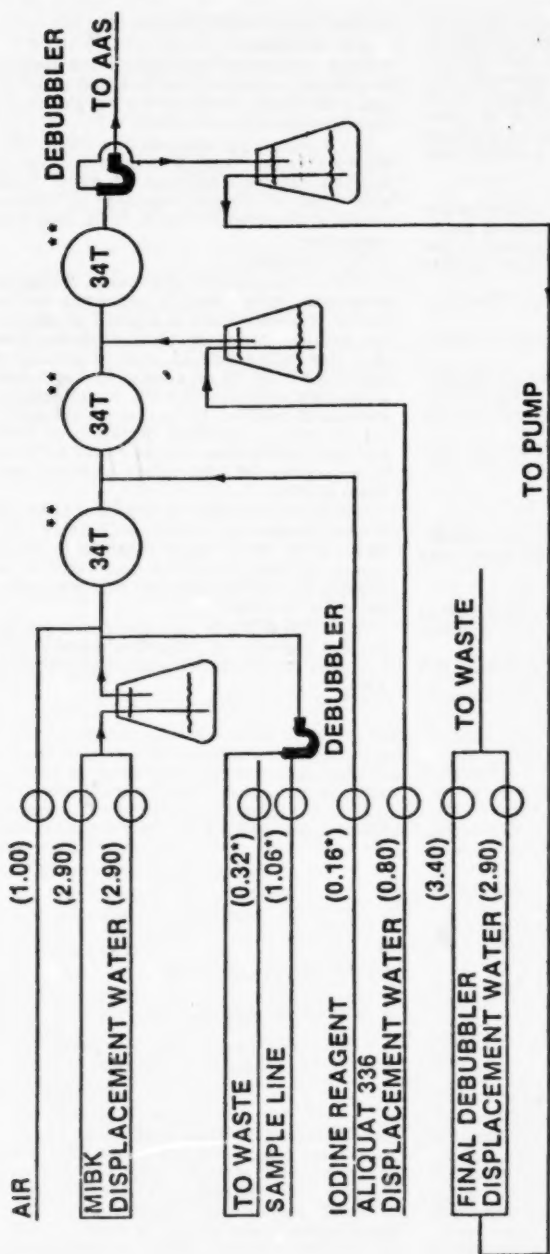


Figure 1

Flow Diagram for the Automated System. (*: Solvaflex pump tubing, **: Technicon part no. 157-0225)
Numbers in parentheses refer to the flow rate (ml/min.).

METHOD 3—TEST FOR LEAD IN GASOLINE BY X-RAY SPECTROMETRY**1. Scope and application.**

1.1 This method covers the determination of the total lead content of gasoline. The procedure's calibration range is 0.010 to 5.0 grams of lead/U.S. gallon. Samples above this level should be diluted to fall within the range of 0.05 to 5.0 grams of lead/U.S. gallon. The method compensates for variations in gasoline composition and is independent of lead alkyl type.

1.2 This method may be used as an alternative to Method 1—Standard Method Test for Lead in Gasoline by Atomic Absorption Spectrometry, or to Method 2—Automated Method Test for Lead in Gasoline by Atomic Absorption Spectrometry.

1.3 Where trade names or specific products are noted in the method, equivalent apparatus and chemical reagents may be used. Mention of trade names or specific products is for the assistance of the user and does not constitute endorsement by the U.S. Environmental Protection Agency.

2. Summary of method.

2.1 A portion of the gasoline sample is placed in an appropriate holder and loaded into an X-ray spectrometer. The ratio of the net X-ray intensity of the lead L alpha radiation to the net intensity of the incoherently scattered tungsten L alpha radiation is measured. The lead content is determined by reference to a linear calibration equation which relates the lead content to the measured ratio.

2.2 The incoherently scattered tungsten radiation is used to compensate for variations in gasoline samples.

3. Sample handling and preservation.

3.1 Samples should be collected and stored in containers which will protect them from changes in the lead content of the gasoline, such as loss of volatile fractions of the gasoline by evaporation or leaching of the lead into the container or cap.

3.2 If samples have been refrigerated they should be brought to room temperature prior to analysis.

3.3 Gasoline is extremely flammable and should be handled cautiously and with adequate ventilation. The vapors are harmful if inhaled and prolonged breathing of vapors should be avoided. Skin contact should be minimized. See precautionary statements in Annex A1.3.

4. Apparatus.

4.1 X-ray Spectrometer, capable of exciting and measuring the fluorescence lines mentioned in 2.1 and of being operated under the following instrumental conditions or others giving equivalent results: a tungsten target tube operated at 50 kV, a lithium fluoride analyzing crystal, an air or

helium optical path and a proportional or scintillation detector.

4.2 Some manufacturers of X-ray Spectrometer units no longer allow use of air as the beam path medium because the X-ray beam produces ozone, which may degrade seals and electronics. In addition, use of the equipment with liquid gasoline in close proximity to the hot X-ray tube could pose flammability problems with any machine in case of a rupture of the sample container. Therefore, use of the helium alternative is recommended.

5. Reagents.

5.1 Isooctane. Isooctane is flammable and the vapors may be harmful. See precautions in Annex A1.1.

5.2 Lead standard solution, in isooctane, toluene or a mixture of these two solvents, containing approximately 5 gm Pb/U.S. gallon may be prepared from a lead-in-oil concentrate such as those prepared by Conostan (Conoco, Inc., Ponca City, Oklahoma). Isooctane and toluene are flammable and the vapors may be harmful. See precautionary statements in Annex A1.1 and A1.2.

6. Calibration.

6.1 Make exact dilutions with isooctane of the lead standard solution to give solutions with concentrations of 0.01, 0.05, 0.10, 0.50, 1.0, 3.0 and 5.0 g Pb/U.S. gallon. If a more limited range is desired as required for linearity, such range shall be covered by at least five standard solutions approximately equally spaced and this range shall not be exceeded by any of the samples. Place each of the standard solutions in a sample cell using techniques consistent with good operating practice for the spectrometer employed. Insert the sample in the spectrometer and allow the spectrometer atmosphere to reach equilibrium (if appropriate). Measure the intensity of the lead L alpha peak at 1.175 angstroms, the Compton scatter peak of the tungsten L alpha line at 1.500 angstroms and the background at 1.211 angstroms. Each measured intensity should exceed 200,000 counts or the time of measurement should be at least 30 seconds. The relative standard deviation of each measurement, based on counting statistics, should be one percent or less. The Compton scatter peak given above is for 90° instrument geometry and should be changed for other geometries. The Compton scatter peak (in angstroms) is found at the wavelength of the tungsten L alpha line plus $0.024(1 - \cos \phi)$, where ϕ is the angle between the incident radiation and the take-off collimator.

6.2 For Each of the standards, as well as for an isooctane blank, determine the net lead intensity by subtracting the corrected background from the gross intensity. Determine the corrected background by multiplying the intensity of the background at 1.211

angstroms by the following ratio obtained on an isooctane blank:

Background at 1.175 angstroms

Background at 1.211 angstroms

6.3 Determine the corrected lead intensity ratio, which is the net lead intensity corrected for matrix effects by division by the net incoherently scattered tungsten radiation. The net scattered intensity is calculated by subtracting the background intensity at 1.211 angstroms from the gross intensity of the incoherently scattered tungsten L alpha peak. The equation for the corrected lead intensity ratio follows:

$$R = \frac{\text{Lead L alpha—corrected background}}{\text{Incoherent tungsten L alpha—background}}$$

6.4 Obtain a linear calibration curve by performing a least squares fit of the corrected lead intensity ratios to the standard concentrations.

7. Procedure.

7.1 Prepare a calibration curve as described in 6. Since the scattered tungsten radiation serves as an internal standard, the calibration curve should serve for at least several days. Each day the suitability of the calibration curve should be checked by analyzing several National Bureau of Standards (NBS) lead-in-reference-fuel standards or other suitable standards.

7.2 Determine the corrected lead intensity ratio for a sample in the same manner as was done for the standards. The samples should be brought to room temperature before analysis.

7.3 Determine the lead concentration of the sample from the calibration curve. If the sample concentration is greater than 5.0 g Pb/U.S. gallon or the range calibrated for in 6.1, the sample should be diluted so that the result is within the calibration span of the instrument.

7.4 Quality control standards, such as NBS standard reference materials, should be analyzed at least once every testing session.

7.5 For each group of ten samples, a spiked sample should be prepared by adding a known amount of lead to a sample. This known addition should be at least 0.05 g Pb/U.S. gallon, at least 50% of the measured lead content of the unspiked sample, and not more than 200% of the measured lead content of the unspiked sample (unless the minimum addition of 0.05 g Pb/U.S. gallon exceeds 200%). Both the spiked and unspiked samples should be analyzed.

8. Quality control.

8.1 The difference between duplicates should not exceed 0.005 g Pb/U.S. gallon or a relative difference of 6%.

8.2 All quality control standard check samples should agree within 10% of the nominal value of the standard.

8.3 All spiked samples should have a percent recovery of $100\% \pm 10\%$. The percent recovery, P, is calculated as follows:

$$P = 100 \times (A-B)/K$$

where

A = the analytical result from the spiked sample, B = the analytical result from the unspiked sample, and K = the known addition.

8.4 The difference between independent analyses of the same sample in different laboratories should not exceed 0.01 g Pb/U.S. gallon or a relative difference of 12%.

9. Past quality control data.

9.1 Duplicate analysis for 26 samples in the range of 0.01 to 0.10 g Pb/U.S. gallon resulted in an average relative difference of 5.2% with a standard deviation of 5.4%. Duplicate analysis of 14 samples in the range 0.1 to 0.5 g Pb/U.S. gallon resulted in an average relative difference of 2.3% with a standard deviation of 2.0. Duplicate analysis of 47 samples in the range of 0.5 to 5 g Pb/U.S. gallon resulted in an average relative difference of 2.1% with a standard deviation of 1.8%.

9.2 The average percent recovery for 23 spikes made to samples in the 0.0 to 0.1 g Pb/U.S. gallon range was 103% with a standard deviation of 3.2%. For 42 spikes made to samples in the 0.1 to 5.0 g Pb/U.S. gallon range, the average percent recovery was 102% with a standard deviation of 4.2%.

9.3 The analysis of National Bureau of Standards lead-in-reference-fuel standards of known concentrations in a single laboratory has resulted in found values deviating from the true value for 14 determinations of 0.0490 g Pb/U.S. gallon by an average of 2.8% with a standard deviation of 6.4%, for 11 determinations of 0.065 g Pb/U.S. gallon by an average of 4.4% with a standard deviation of 2.9%, and for 15 determinations of 1.994 g Pb/U.S. gallon by an average of 0.3% with a standard deviation of 1.3%.

9.4 Eighteen analyses of reference samples (U.S. EPA, Research Triangle Park, NC) have resulted in found values differing from the true value by an average of 0.0004 g Pb/U.S. gallon with a standard deviation of 0.004 g Pb/U.S. gallon.

ANNEX

A1. Precautionary Statements

A1.1 Isooctane

Danger—Extremely flammable. Vapors harmful if inhaled.
Vapor may cause flash fire.
Keep away from heat, sparks, and open flame.
Vapors are heavier than air and may gather in low places, resulting in explosion hazard.
Keep container closed.
Use adequate ventilation.
Avoid buildup of vapors.
Avoid prolonged breathing of vapor or spray mist.
Avoid prolonged or repeated skin contact.

A1.2 Toluene

Warning—Flammable. Vapor harmful.*
Keep away from heat, sparks, and open flame.
Keep container closed.
Use with adequate ventilation.
Avoid breathing of vapor or spray mist.
Avoid prolonged or repeated contact with skin.

A1.3 Gasoline

Danger—Extremely flammable. Vapors harmful if inhaled.
Vapor may cause flash fire.
Keep away from heat, sparks, and open flame.
Vapors are heavier than air and may gather in low places, resulting in explosion hazard.
Keep container closed.
Use adequate ventilation.
Avoid buildup of vapors.
Avoid prolonged breathing of vapor or spray mist.
Avoid prolonged or repeated skin contact.

[39 FR 24891, July 8, 1974; 39 FR 25653, July 12, 1974; 39 FR 26287, July 18, 1974, as amended at 47 FR 765, Jan. 7, 1982; 52 FR 159, Jan. 5, 1987]

APPENDIX C—LIST OF COUNTIES WITH POPULATION DENSITIES LESS THAN 50 PERSONS PER SQUARE MILE (EXCLUDING CITIES WITH POPULATIONS EQUAL TO OR GREATER THAN 50,000 PERSONS)

State/County	Population ¹
Alaska:	
Aleutians East	41
Aleutians West	38
Barrow	25

State/County	Population ¹
Bibb	22
Blount	42
Bullock	19
Butler	28
Cherokee	28
Chilton	36
Choctaw	18
Clarke	22
Clay	21
Cleburne	19
Conecuh	18
Coosa	16
Covington	35
Creshaw	22
Escambia	36
Fayette	26
Franklin	37
Geneva	38
Greene	17
Hale	24
Henry	24
Jackson	36
La Mar	24
Lawrence	40
Lowndes	18
Macon	40
Marengo	24
Marion	32
Monroe	20
Montgomery	46
Perry	21
Pickens	23
Pike	37
Randolph	32
St. Clair	44
Selby	48
Sumter	19
Tallapoosa	48
Tuscaloosa	39
Washington	15
Wilcox	18
Winston	27
Alaska:	
Aleutian Islands	1
Angeon	1
Barrow	1
Bethel	1
Bristol Bay Borough	2
Bristol Bay	1
Cordova-McCarthy	1
Fairbanks	6
Haines	1
Juneau	11
Kenai-Cook Inlet	1
Ketchikan	7
Kobuk	1
Kodiak	2
Kuskokwim	1
Matanuska-Susitna	1
Nome	1
Outer Ketchikan	1
Prince of Wales	1
Seward	1
Sitka	3
Skagway-Yakutat	1
Southeast Fairbanks	1
Upper Yukon	1
Valdez-Chitina-Whittier	1
Wade Hampton	1
Wrangell-Petersburg	1
Yukon-Koyukuk	1
Arizona:	
Apache	3

State/County	Population ¹	State/County	Population ¹
Cochise.....	10	Sharp.....	14
Coconino.....	3	Stone.....	11
Gila.....	6	Union.....	43
Graham.....	4	Van Buren.....	12
Greenlee.....	5	White.....	38
Maricopa.....	22	Woodruff.....	20
Mohave.....	2	Yell.....	15
Navajo.....	5	California:	
Pima.....	10	Alpine.....	1
Pinal.....	13	Amador.....	20
Santa Cruz.....	11	Calaveras.....	13
Yavapai.....	5	Colusa.....	11
Yuma.....	6	Del Norte.....	14
Arkansas:		El Dorado.....	26
Arkansas.....	23	Fresno.....	42
Ashley.....	27	Gienn.....	13
Baxter.....	29	Humboldt.....	28
Bacon.....	33	Imperial.....	16
Bradley.....	20	Inyo.....	2
Calhoun.....	9	Kern.....	32
Carroll.....	20	Kings.....	48
Chicot.....	28	Lake.....	16
Clark.....	25	Lassen.....	4
Clay.....	29	Madera.....	19
Cleburne.....	19	Mariposa.....	4
Cleveland.....	11	Mendocino.....	15
Columbia.....	11	Modoc.....	2
Columbia.....	34	Mono.....	1
Conway.....	30	Nevada.....	27
Crawford.....	43	Plumas.....	5
Cross.....	32	Riverside.....	45
Dallas.....	15	San Benito.....	13
Desha.....	25	San Bernardino.....	26
Drew.....	18	San Luis Obispo.....	33
Faulkner.....	49	Shasta.....	20
Franklin.....	18	Sierra.....	2
Fulton.....	13	Siskiyou.....	5
Grant.....	15	Tehama.....	10
Greene.....	43	Trinity.....	2
Hempstead.....	27	Tulare.....	39
Hot Springs.....	35	Tuolumne.....	10
Howard.....	20	Colorado:	
Independence.....	30	Alamosa.....	16
Izard.....	13	Archuleta.....	2
Jackson.....	33	Baca.....	2
Jefferson.....	32	Bent.....	4
Johnson.....	20	Chaffee.....	10
Lafayette.....	19	Cheyenne.....	1
Lawrence.....	28	Clear Creek.....	12
Lee.....	31	Conejos.....	6
Lincoln.....	23	Costilla.....	3
Little River.....	23	Crowley.....	4
Logan.....	23	Custer.....	2
Lonoke.....	33	Delta.....	13
Madison.....	11	Dolores.....	2
Manion.....	12	Douglas.....	10
Monroe.....	26	Eagle.....	4
Montgomery.....	8	Elbert.....	2
Nevada.....	16	El Paso.....	46
Newton.....	7	Fremont.....	14
Quachita.....	42	Garfield.....	5
Perry.....	10	Gilpin.....	9
Pike.....	15	Grand.....	2
Poinsett.....	35	Gunnison.....	2
Polk.....	15	Hinsdale.....	1
Pope.....	35	Huerfano.....	4
Prairie.....	16	Jackson.....	1
Randolph.....	20	Kiowa.....	3
St. Francis.....	49	Kit Carson.....	20
Scott.....	9	Lake.....	11
Searcy.....	12	La Plata.....	34
Sebastian.....	34	Lanier.....	3
Sevier.....	22	Las Animas.....	

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State/County	Population ¹
Lincoln	2
Logan	10
Mesa	16
Mineral	1
Moffat	1
Montezuma	6
Montrose	8
Morgan	16
Otero	19
Oursy	3
Park	1
Phillips	6
Pitkin	6
Powers	8
Rio Blanco	1
Rio Grande	11
Routt	3
Saguache	1
San Juan	2
San Miguel	2
Sedgwick	6
Summit	4
Teller	6
Washington	2
Weld	22
Yuma	4
Florida	
Alachua	45
Baker	16
Calhoun	14
Charlotte	39
Citrus	34
Collier	19
Columbia	32
De Soto	20
Doe	8
Flagler	9
Franklin	13
Gilchrist	10
Glades	5
Gulf	18
Hamilton	15
Hardee	24
Hendry	10
Hernando	35
Highlands	30
Holmes	22
Jackson	37
Jefferson	15
Lafayette	5
Leon	48
Levy	12
Liberty	4
Madison	19
Manatee	43
Nassau	32
Okeechobee	14
Osceola	19
Putnam	47
Santa Rosa	37
Sumter	27
Suwannee	23
Taylor	13
Union	34
Wakulla	10
Walton	15
Washington	20
Georgia	
Appling	25
Atkinson	18
Bacon	28
Baker	11
Banks	30

State/County	Population ¹
Barnes	25
Bleckley	47
Brantley	13
Brooks	28
Bryan	15
Bullock	46
Burke	22
Calhoun	23
Camden	17
Candler	26
Charlton	7
Clay	18
Clinch	8
Coffee	37
Crawford	18
Dawson	17
Decatur	39
Dodge	31
Dooly	26
Early	24
Echols	5
Effingham	28
Elbert	48
Emanuel	27
Evans	39
Fannin	34
Franklin	49
Gilmer	20
Glascock	16
Grady	38
Greene	25
Hancock	19
Harris	25
Heard	18
Irwin	22
Jasper	15
Jeff Davis	28
Jefferson	32
Jenkins	24
Johnson	25
Jones	30
Lanier	28
Laurens	40
Lee	20
Liberty	34
Lincoln	31
Long	9
Lumpkin	30
McIntosh	17
Macon	32
Madison	46
Marion	14
Meriwether	39
Miller	22
Mitchell	37
Monroe	28
Montgomery	26
Morgan	28
Murray	38
Oconee	43
Oglethorpe	17
Pickens	43
Pierce	27
Pike	32
Pulaski	32
Putnam	25
Quitman	14
Rabun	23
Randolph	20
Schley	19
Screven	19
Seminole	29
Stewart	14

State/County	Population ¹	State/County	Population ¹
Talbot.....	17	Bureau.....	45
Taliaferro.....	12	Calhoun.....	23
Tattnall.....	34	Carroll.....	42
Taylor.....	20	Cass.....	38
Telfair.....	26	Clark.....	32
Terrell.....	35	Clay.....	32
Towns.....	28	Crawford.....	45
Treutlen.....	29	Cumberland.....	28
Turner.....	30	De Witt.....	43
Twiggs.....	23	Douglas.....	45
Union.....	22	Edgar.....	34
Ware.....	37	Edwards.....	32
Warren.....	23	Fayette.....	30
Washington.....	26	Ford.....	34
Wayne.....	28	Fulton.....	48
Webster.....	12	Gallatin.....	23
Wheeler.....	15	Greene.....	31
White.....	32	Hamilton.....	20
Wilcox.....	18	Hancock.....	30
Wilkes.....	22	Hardin.....	27
Wilkinson.....	21	Henderson.....	22
Worth.....	26	Hoquiam.....	30
Hawaii:		Jasper.....	22
Hawaii.....	16	Jersey.....	49
Kauai.....	48	Jo Daviess.....	36
Maui.....	39	Johnson.....	22
Idaho:		Lawrence.....	47
Ada.....	37	Livingston.....	36
Adams.....	2	Marshall.....	34
Bannock.....	47	Mason.....	30
Bear Lake.....	6	Menard.....	21
Benewah.....	8	Mercer.....	31
Bingham.....	14	Monroe.....	49
Blaine.....	2	Montgomery.....	43
Borise.....	1	Moultrie.....	41
Bonner.....	9	Perry.....	45
Bonneville.....	29	Piatt.....	25
Boundary.....	4	Pike.....	23
Butte.....	1	Pope.....	10
Camas.....	1	Pulaski.....	43
Caribou.....	4	Putnam.....	31
Cassia.....	7	Richland.....	46
Clark.....	1	Schuyler.....	19
Clearwater.....	4	Scott.....	24
Custer.....	1	Shelby.....	30
Elmore.....	6	Stark.....	28
Franklin.....	11	Union.....	39
Fremont.....	5	Warren.....	40
Gem.....	17	Washington.....	24
Gooding.....	12	Wayne.....	24
Idaho.....	2	White.....	34
Jefferson.....	11	Indiana:	
Jerome.....	17	Benton.....	28
Kootenai.....	28	Brown.....	28
Latah.....	23	Carroll.....	47
Lemhi.....	1	Crawford.....	26
Lewis.....	8	Fountain.....	46
Lincoln.....	3	Franklin.....	43
Madison.....	28	Fulton.....	46
Minidoka.....	21	Greene.....	49
Nez Perce.....	36	Harrison.....	43
Oneida.....	2	Jasper.....	36
Owyhee.....	1	Martin.....	32
Payette.....	31	Newton.....	28
Power.....	3	Ohio.....	49
Shoshone.....	8	Orange.....	42
Teton.....	5	Owen.....	31
Twin Falls.....	21	Parke.....	33
Valley.....	1	Pike.....	37
Washington.....	5	Pulaski.....	29
Illinois:		Ripley.....	48
Bond.....	37	Spencer.....	17
Brown.....	18	Sullivan.....	44

State/County	Population ¹	State/County	Population ¹
Switzerland.....	29	Humboldt.....	29
Union.....	39	Ida.....	22
Warren.....	24	Iowa.....	26
Washington.....	37	Jackson.....	32
White.....	42	Jasper.....	48
Wis.		Jefferson.....	36
Adair.....	17	Jones.....	34
Adams.....	15	Keokuk.....	24
Allamakee.....	24	Kossuth.....	23
Appanoose.....	29	Louisa.....	27
Audubon.....	21	Lucas.....	23
Benton.....	32	Lyon.....	23
Boone.....	46	Madison.....	20
Buchanan.....	38	Mahaska.....	39
Buena Vista.....	36	Mills.....	26
Butler.....	29	Mitchell.....	28
Calhoun.....	25	Monona.....	17
Carroll.....	40	Monroe.....	22
Cass.....	30	Montgomery.....	30
Cedar.....	30	O'Brien.....	30
Cherokee.....	30	Osceola.....	21
Chickasaw.....	30	Page.....	35
Clarke.....	18	Palo Alto.....	24
Clay.....	32	Plymouth.....	28
Clayton.....	26	Pocahontas.....	22
Crawford.....	27	Pottawattamie.....	29
Dallas.....	44	Poweshiek.....	32
Davis.....	16	Ringgold.....	12
Decatur.....	18	Sac.....	27
Delaware.....	33	Shelby.....	26
Dickinson.....	33	Sioux.....	37
Dubuque.....	46	Tama.....	28
Emmet.....	36	Taylor.....	17
Effingham.....	28	Union.....	32
Elbert.....	48	Van Buren.....	18
Emanuel.....	27	Warren.....	49
Evans.....	39	Washington.....	33
Fennell.....	34	Wayne.....	16
Franklin.....	49	Winnebago.....	32
Gilmer.....	20	Winneshek.....	32
Glascock.....	16	Woodbury.....	21
Grady.....	38	Worth.....	22
Greene.....	25	Kansas:	
Hancock.....	19	Allen.....	30
Harris.....	25	Anderson.....	15
Harold.....	18	Atchison.....	45
Iowa.....	22	Barber.....	6
Jasper.....	15	Barton.....	34
Jeff Davis.....	28	Bourbon.....	24
Jefferson.....	32	Brown.....	20
Jenkins.....	24	Butler.....	27
Johnson.....	25	Chase.....	4
Jones.....	30	Chautauqua.....	7
Lamar.....	28	Cherokee.....	37
Laurens.....	40	Cheyenne.....	4
Lee.....	20	Clark.....	3
Liberty.....	34	Clay.....	16
Lincoln.....	31	Cloud.....	19
Long.....	9	Coffey.....	12
Lumpkin.....	30	Comanche.....	3
Fayette.....	37	Cowley.....	31
Floyd.....	39	Decatur.....	6
Franklin.....	23	Dickinson.....	23
Fremont.....	18	Doniphan.....	23
Greene.....	22	Edwards.....	7
Grundy.....	28	Elk.....	6
Guthrie.....	21	Ellis.....	27
Hamilton.....	32	Ellsworth.....	9
Hancock.....	24	Finney.....	15
Hardin.....	39	Ford.....	21
Harrison.....	23	Franklin.....	35
Henry.....	41	Gove.....	4
Howard.....	24	Graham.....	5

State/County	Population ¹	State/County	Population ¹
Grant	10	Casey	30
Gray	5	Clay	39
Greeley	2	Clinton	43
Greenwood	8	Crittenden	23
Hamilton	3	Cumberland	22
Harper	10	Elliott	29
Haskell	6	Estill	25
Hodgeman	3	Fleming	32
Jackson	16	Gallatin	41
Jefferson	23	Garrard	40
Jewell	7	Grant	40
Kearney	4	Grayson	33
Kingman	10	Green	37
Kiowa	6	Hancock	38
Labette	39	Harrison	46
Lane	4	Hart	33
Lin.	13	Henry	38
Logan	4	Hickman	25
Lyon	38	Jackson	30
McPherson	28	Knott	41
Manon	15	Larue	41
Marshall	15	Lee	25
Meade	5	Leslie	31
Miami	33	Letcher	28
Mitchell	11	Lewis	26
Morris	9	Lincoln	46
Morton	5	Livingston	24
Nemaha	17	Logan	39
Neosho	32	Lyon	26
Ness	8	McCreary	30
Norton	8	McLean	35
Osage	19	Magoffin	34
Osborne	7	Manon	46
Ottawa	9	Martin	41
Pawnee	11	Menfee	19
Phillips	9	Metcalfe	28
Pottawatomie	14	Monroe	35
Pratt	14	Morgan	27
Rawlins	4	Nicolas	32
Reno	48	Ohio	32
Republic	12	Owen	21
Rice	17	Owsley	25
Rooks	9	Pendleton	36
Rush	7	Powell	45
Russell	11	Robertson	21
Scott	8	Rockcastle	40
Seward	25	Russell	44
Shendan	4	Spencer	28
Sherman	7	Todel	29
Smith	8	Trigg	21
Stafford	7	Trimble	37
Stevens	6	Union	47
Sumner	20	Washington	35
Thomas	7	Wayne	32
Trego	5	Webster	39
Wabaunsee	8	Wolfe	25
Wallace	2		
Washington	10	Louisiana:	
Wichita	5	Allen	27
Wilson	20	Avoyelles	45
Woodson	10	Beauregard	19
		Bienville	19
Kentucky:		Caldwell	17
Adair	35	Cameron	6
Allen	36	Catahoula	16
Anderson	45	Claiborne	22
Ballard	32	Concordia	31
Bath	32	De Soto	25
Bracken	35	East Carroll	30
Breathitt	29	East Feliciana	39
Breckinridge	27	Evangeline	46
Butler	22	Franklin	37
Caldwell	37	Grant	20
Carlisle	27	Iberville	49

Environmental Protection Agency

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State/County	Population ¹	State/County	Population ¹
Jackson	27	Otsego	20
Jefferson Davis	45	Presque Isle	20
La Salle	21	Roscommon	19
Madison	23	Sanilac	37
Morehouse	40	Schoolcraft	7
Natchitoches	27	Welford	35
Plaquemines	24	Minnesota	
Pontie Coupee	39	Artkin	6
Red River	23	Becker	19
Richland	38	Beltrami	11
Sabine	21	Big Stone	16
St. Helena	24	Brown	47
St. Martin	44	Carlton	33
Tensas	16	Cass	9
Union	21	Chippewa	26
Vermilion	36	Chisago	42
Vernon	40	Clay	45
West Carroll	37	Clearwater	8
West Feliciana	28	Cook	3
Winn	17	Cottonwood	23
Mane		Crow Wing	35
Aroostook	14	Dodge	30
Franklin	13	Douglas	35
Hancock	23	Fairbault	29
Lincoln	45	Falmore	26
Oxford	21	Goodhue	46
Penobscot	37	Grant	14
Piscataquis	4	Houston	31
Somerset	10	Hubbard	11
Waldo	32	Isanti	38
Washington	12	Itasca	13
Maryland		Jackson	21
Garrett	33	Kanabec	19
Queen Annes	49	Kandiyohi	39
Michigan		Kittson	6
Alcona	10	Koochiching	5
Alger	9	Lac Qui Parle	15
Antrim	26	Lake	6
Arenac	30	Lake of the Woods	3
Baraga	9	Le Sueur	48
Benzie	27	Lincoln	15
Charlevoix	40	Lyon	34
Cheboygan	23	Mahnomen	10
Chippewa	20	Marshall	7
Clare	29	Martin	35
Crawford	12	Meeker	30
Delta	31	Millie Lacs	28
Dickinson	31	Morrison	24
Emmet	40	Murray	18
Gladwin	27	Nobles	33
Gogebic	19	Norman	11
Houghton	34	Otter Tail	23
Huron	42	Pennington	21
Iosco	46	Pine	12
Iron	12	Pipestone	28
Kalkaska	9	Polk	17
Keweenaw	4	Pope	17
Lake	10	Red Lake	12
Leelanau	32	Redwood	23
Luce	7	Renville	22
Mackinac	10	Rock	23
Manistee	37	Roseau	7
Marquette	35	St. Louis	36
Mason	46	Sherburne	43
Menominee	24	Sibley	27
Missaukee	13	Stevens	20
Montmorency	9	Swift	18
Newaygo	33	Todd	23
Oceana	34	Traverse	11
Ogemaw	21	Wabasha	33
Ontonagon	8	Wadena	23
Osceola	26	Waseca	40
Oscoda	8	Watsonwan	31

State/County	Population ¹
Wilkin	12
Yellow Medicine	19
Mississippi	
Amite	19
Attala	27
Benton	18
Calhoun	25
Carroll	15
Chickasaw	33
Choctaw	20
Claborne	21
Clarke	22
Clay	46
Copiah	32
Covington	34
Franklin	14
George	26
Greene	12
Grenada	46
Hancock	36
Holmes	30
Humphreys	35
Issaquena	7
Itawamba	31
Jasper	23
Jefferson	18
Jefferson Davis	31
Kemper	14
Lafayette	36
Lamar	30
Lawrence	26
Leake	29
Lincoln	45
Madison	41
Marion	42
Marshall	34
Monroe	44
Montgomery	32
Neshoba	37
Newton	33
Noxubee	21
Panola	39
Pearl River	34
Perry	14
Pontotoc	35
Prentiss	48
Quitman	39
Scott	35
Sharkey	20
Simpson	34
Smith	21
Stone	18
Tallahatchie	30
Tate	46
Tippah	34
Thomson	34
Tunica	26
Union	45
Walthall	31
Wayne	20
Webster	24
Wilkinson	16
Winston	30
Yalobusha	24
Yazoo	29
Missouri	
Adair	39
Andrew	27
Atchison	17
Audrain	37
Barry	25
Barton	18
Bates	18

State/County	Population ¹
Benton	13
Bollinger	14
Butler	47
Caldwell	19
Callaway	31
Camden	21
Carroll	18
Carter	8
Cedar	19
Chariton	15
Christian	27
Clark	16
Clinton	30
Cooper	26
Crawford	20
Dade	14
Dallas	19
Davies	15
Dekalb	17
Dent	15
Douglas	11
Gasconade	23
Gentry	17
Grundy	27
Harrison	14
Henry	25
Hickory	12
Holt	15
Howard	22
Howell	26
Iron	17
Johnson	41
Knox	11
Laclede	26
Lafayette	42
Lawrence	40
Lewis	22
Lincoln	29
Linn	24
Livingston	29
McDonald	23
Macon	19
Madison	17
Manes	13
Mercer	11
Miller	25
Mississippi	40
Moniteau	26
Monroe	14
Montgomery	21
Morgan	17
New Madrid	34
Nodaway	26
Oregon	12
Osage	18
Ozark	9
Perry	31
Phelps	44
Pike	25
Polk	24
Putnam	16
Ralls	47
Randolph	31
Ray	7
Reynolds	15
Ripley	11
St. Clair	26
Ste. Genevieve	33
Saline	15
Schuyler	12
Scotland	7
Shannon	16
Shelby	

State/County	Population ¹
Stoddard	31
Stone	22
Sullivan	12
Taney	21
Texas	15
Vernon	23
Warren	23
Washington	20
Wayne	11
Webster	26
Worth	13
Wright	20
Montana:	
Beaverhead	1
Big Horn	2
Blaine	2
Broadwater	2
Carbon	3
Carter	1
Cascade	8
Chouteau	2
Custer	3
Danels	2
Dawson	5
Deer Lodge	21
Fallon	2
Fergus	3
Flathead	8
Gallatin	13
Garfield	0
Glacier	4
Golden Valley	1
Granite	2
Hill	6
Jefferson	3
Judith Basin	1
Lake	10
Lewis and Clark	10
Liberty	2
Lincoln	5
McCone	1
Madison	1
Meagher	1
Mineral	2
Missoula	22
Musselshell	2
Park	4
Petroleum	0
Philps	1
Pondera	4
Powder River	1
Powell	3
Prairie	1
Ravalli	6
Richland	5
Roosevelt	4
Rosebud	1
Sanders	3
Shenando	3
Stillwater	3
Sweet Grass	2
Teton	3
Toole	3
Treasure	1
Valley	2
Wheatland	2
Wibaux	2
Yellowstone	10
Nebraska:	
Antelope	11
Arthur	1
Banner	1
Blaine	1

State/County	Population ¹
Boone	12
Box Butte	9
Boyd	7
Brown	3
Buffalo	33
Burt	19
Butler	16
Cass	33
Cedar	16
Chase	5
Cherry	1
Cheyenne	9
Clay	15
Colfax	23
Cuming	21
Custer	6
Dawes	7
Dawson	20
Deuel	6
Dixon	16
Dundy	3
Fillmore	14
Franklin	8
Frontier	4
Furnas	10
Gage	30
Garden	2
Garfield	4
Gosper	5
Grant	1
Greeley	7
Hamilton	7
Harlan	8
Hayes	2
Hitchcock	6
Holt	5
Hooker	1
Howard	12
Jefferson	18
Johnson	15
Kearney	13
Keith	8
Keya Paha	2
Kimball	6
Knox	11
Lancaster	23
Lincoln	12
Logan	2
Loup	1
McPherson	1
Madison	48
Merrick	18
Morrill	4
Nance	12
Nemaha	22
Nuckolls	13
Otoe	25
Pawnee	10
Perkins	4
Phelps	18
Pierce	15
Platte	40
Polk	15
Red Willow	18
Richardson	22
Rock	2
Saline	22
Saunders	22
Seward	25
Shenando	3
Sherman	8
Sioux	1
Stanton	13

State/County	Population ¹	State/County	Population ¹
Thayer.....	13	Anson.....	46
Thomas.....	1	Beaufort.....	44
Thurston.....	18	Berrie.....	29
Valley.....	10	Bladen.....	30
Washington.....	34	Brunswick.....	28
Wayne.....	23	Camden.....	23
Webster.....	9	Caswell.....	45
Wheeler.....	2	Chatham.....	42
York.....	24	Cherokee.....	36
Nevada:		Clay.....	25
Churchill.....	2	Cumtuck.....	28
Clark.....	19	Dare.....	18
Douglas.....	10	Duplin.....	47
Elko.....	1	Gates.....	25
Esmeralda.....	0	Graham.....	22
Eureka.....	0	Greene.....	26
Humboldt.....	1	Hoke.....	42
Lander.....	0	Hyde.....	9
Lincoln.....	0	Jackson.....	44
Lyon.....	4	Jones.....	21
Mineral.....	2	Macon.....	31
Nye.....	0	Madison.....	36
Pershing.....	0	Montgomery.....	39
Storey.....	3	Northampton.....	45
Washoe.....	6	Pamlico.....	28
White Pine.....	1	Pender.....	21
New Hampshire:		Perquimans.....	34
Carroll.....	20	Polk.....	49
Coos.....	19	Sampson.....	48
Grafton.....	32	Swain.....	17
New Mexico:		Tyrell.....	10
Catron.....	0	Warren.....	37
Chaves.....	7	Washington.....	41
Colfax.....	3	Yancey.....	40
Curry.....	28	North Dakota:	
De Baca.....	1	Adams.....	4
Dona Ana.....	18	Barnes.....	10
Eddy.....	10	Benson.....	6
Grant.....	6	Billings.....	1
Guadalupe.....	2	Bottineau.....	6
Harding.....	1	Bowman.....	3
Hidalgo.....	1	Burke.....	4
Lea.....	11	Burleigh.....	25
Lincoln.....	2	Cass.....	12
Luna.....	4	Cavalier.....	5
McKinley.....	8	Dickey.....	6
Mora.....	2	Divide.....	4
Otero.....	6	Dunn.....	2
Quay.....	4	Eddy.....	6
Rio Arriba.....	4	Emmons.....	5
Roosevelt.....	7	Foster.....	7
Sandoval.....	5	Golden Valley.....	3
San Juan.....	10	Grand Forks.....	42
San Miguel.....	5	Grant.....	3
Santa Fe.....	29	Griggs.....	6
Sierra.....	2	Hettinger.....	4
Socorro.....	1	Kidder.....	3
Taos.....	8	Lamoure.....	6
Torrance.....	2	Logan.....	4
Union.....	1	McHenry.....	5
Valencia.....	7	McIntosh.....	6
New York:		McKenzie.....	2
Allegany.....	44	McLean.....	5
Delaware.....	31	Mercer.....	6
Essex.....	19	Morton.....	11
Franklin.....	26	Mountrail.....	5
Hamilton.....	3	Melton.....	6
Herkimer.....	47	Oiver.....	3
Lewis.....	18	Pembina.....	10
St. Lawrence.....	41	Pierce.....	6
Schoharie.....	40	Ramsey.....	10
North Carolina:		Ransom.....	8
Alleghany.....	36	Renville.....	4

Environmental Protection Agency

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State/County	Population ¹
Richland	12
Rollette	13
Sargent	7
Shendan	3
Sioux	3
Slope	1
Stark	15
Steele	5
Stutsman	10
Towner	4
Trail	11
Walsh	13
Ward	29
Wells	6
Williams	9
Ohio:	
Adams	32
Harrison	42
Hocking	48
Megs	45
Monroe	35
Morgan	29
Noble	26
Paulding	46
Pike	43
Vinton	23
Oklahoma:	
Adair	27
Alfalfa	8
Aloka	11
Beaver	4
Beckham	17
Blaine	13
Bryan	29
Caddo	23
Canadian	36
Carter	45
Cherokee	31
Choctaw	19
Cimarron	2
Coal	11
Comanche	32
Cotton	10
Craig	19
Creek	49
Custer	23
Delaware	25
Dewey	6
Ellis	4
Garvin	31
Grady	27
Grant	7
Greer	13
Harman	9
Harper	5
Haskell	16
Hughes	16
Jackson	38
Jefferson	9
Johnston	12
Kingfisher	14
Kiowa	12
Latimer	12
Le Flore	21
Lincoln	20
Logan	26
Coal	11
Comanche	32
Cotton	10
Craig	19
Creek	49
Custer	23
Delaware	25

State/County	Population ¹
Dewey	6
Ellis	4
Garvin	31
Grady	27
Grant	7
Greer	13
Harman	9
Harper	5
Haskell	16
Hughes	16
Jackson	38
Jefferson	9
Johnston	12
Kingfisher	14
Kiowa	12
Latimer	12
Le Flore	21
Lincoln	20
Logan	26
Love	11
McClain	25
McCurtain	16
McIntosh	21
Major	8
Marshall	21
Maves	36
Murray	25
Noble	14
Nowata	18
Okfuskee	17
Osage	13
Pawnee	20
Pittsburg	30
Pontotoc	39
Pushmataha	7
Roger Mills	4
Rogers	41
Seminole	40
Sequoyah	34
Stephens	40
Texas	8
Tillman	14
Wagoner	39
Washita	12
Woods	9
Woodward	12
Oregon:	
Baker	5
Clatsop	35
Columbia	45
Coos	35
Crook	3
Curry	8
Deschutes	10
Douglas	14
Gilliam	2
Grant	2
Harney	1
Hood River	25
Jackson	34
Jefferson	5
Josephine	22
Klamath	8
Lake	1
Lane	30
Lincoln	26
Linn	31
Malheur	2
Morrow	2
Polk	48
Sherman	3
Tillamook	16
Umatilla	14

State/County	Population ¹	State/County	Population ¹
Union	10	Hyde	3
Wallowa	2	Jackson	2
Warsco	8	Jerauld	6
Wheeler	1	Jones	2
Pennsylvania:		Kingsbury	9
Bedford	42	Lake	20
Cameron	18	Lawrence	22
Clinton	42	Lincoln	20
Elk	47	Lyman	2
Forest	12	McCook	13
Huntingdon	44	McPherson	4
Juniata	43	Marshall	7
Pike	22	Meade	5
Potter	15	Melleite	2
Sulivan	12	Miner	8
Susquehanna	41	Minnehaha	29
Tioga	35	Moody	15
Wayne	40	Pennington	21
Wyoming	48	Perkins	2
South Carolina:		Potter	5
Abbeville	42	Roberts	11
Allendale	23	Sanborn	8
Bamberg	40	Shannon	4
Barnwell	31	Spink	7
Calhoun	30	Stanley	2
Chesterfield	43	Sully	2
Clarendon	43	Todd	5
Colleton	26	Tripp	5
Edgefield	33	Turner	18
Fairfield	29	Union	21
Georgetown	41	Walworth	11
Hampton	28	Washabaugh	1
Jasper	18	Yankton	37
Kershaw	44	Ziebach	1
Lee	45	Tennessee:	
McCormick	22	Benton	31
Newberry	46	Bledsoe	19
Saluda	32	Cannon	31
Williamsburg	37	Carroll	43
South Dakota:		Cheatham	43
Aurora	6	Chester	35
Beadle	3	Claborn	44
Bennett	15	Clay	28
Bon Homme	15	Cumberland	31
Brookings	28	Decatur	28
Brown	22	DeKalb	40
Brule	7	Dickson	45
Ruffalo	4	Fayette	32
Butte	3	Fentress	25
Campbell	4	Franklin	48
Charles Mix	0	Giles	36
Clark	6	Granger	49
Clay	32	Grundy	30
Codington	28	Hancock	29
Corson	2	Hardeman	34
Custer	3	Hardin	31
Davison	40	Haywood	38
Day	8	Henderson	34
Deuel	9	Henry	42
Dewey	2	Hickman	20
Douglas	11	Houston	29
Edmunds	5	Humphreys	26
Fall River	4	Jackson	25
Faulk	4	Johnson	38
Grant	13	Lake	48
Gregory	7	Lauderdale	42
Haakon	2	Lawrence	46
Hamlin	11	Lewis	24
Hand	4	Lincoln	42
Hanson	9	McNairy	32
Harding	1	Macon	41
Hughes	16	Marion	41
Hutchinson	13	Marshall	46

State/County	Population ¹
Meigs	27
Monroe	36
Moore	29
Morgan	25
Overton	34
Perry	13
Pickett	24
Polk	27
Scott	27
Sequatchie	23
Sevier	47
Smith	39
Stewart	16
Trousdale	45
Union	43
Van Buren	15
Wayne	17
White	43
Texas:	
Anderson	26
Andrews	7
Aransas	32
Archer	6
Armstrong	2
Atascosa	16
Austin	21
Bailey	10
Bandera	6
Bastrop	19
Baylor	6
Bee	27
Blanco	5
Borden	1
Bosque	11
Brewster	1
Briscoe	3
Brooks	9
Brown	28
Burleson	15
Burnet	11
Caldwell	39
Calhoun	34
Callahan	10
Camp	42
Carson	7
Cass	26
Castro	12
Chambers	20
Cherokee	31
Childress	9
Clay	7
Cochran	7
Coke	8
Coleman	8
Collingsworth	5
Colorado	19
Comal	43
Comanche	13
Concho	3
Cooke	26
Coryell	34
Cottle	4
Crane	5
Crockett	1
Crosby	10
Culberson	1
Dallam	4
Dawson	18
Deaf Smith	13
Delta	18
De Witt	21
Dickens	4
Dimmit	7

State/County	Population ¹
Donley	4
Duval	6
Eastland	19
Ector	15
Edwards	1
El Paso	39
Erath	17
Falls	23
Fannin	25
Fayette	19
Fisher	7
Floyd	11
Foard	3
Franklin	18
Freestone	13
Frio	10
Gaines	8
Garza	6
Gillespie	10
Glasscock	1
Gonzales	16
Gray	29
Gnines	15
Guadalupe	47
Hale	35
Hall	7
Hamilton	9
Hansford	7
Hardeman	10
Hardin	33
Hartley	2
Haskell	10
Hays	43
Hamphill	3
Henderson	28
Hill	22
Hockley	22
Hood	15
Hopkins	26
Houston	14
Howard	41
Hudspeth	1
Hutchinson	28
Inon	1
Jack	7
Jackson	15
Jasper	27
Jeff Davis	1
Jim Hogg	4
Jim Wells	39
Jones	17
Karnes	18
Kaufman	40
Kendall	10
Kenedy	0
Kent	2
Kerr	18
Kimble	3
King	0
Kinney	1
Kleberg	39
Knox	7
Lamar	40
Lamb	17
Lampasas	13
La Salle	3
Lavaca	18
Lee	13
Leon	8
Liberty	28
Limestone	19
Lipscomb	4
Live Oak	6

State/County	Population ¹	State/County	Population ¹
Llano.....	7	Travis.....	46
Loving.....	0	Trinity.....	11
Lubbock.....	37	Tyler.....	14
Lynn.....	10	Upshur.....	38
McCulloch.....	8	Upton.....	4
McMullen.....	1	Uvalde.....	11
Madison.....	16	Vai Verde.....	8
Marion.....	22	Van Zandt.....	26
Martin.....	5	Walker.....	35
Mason.....	4	Waller.....	28
Matagorda.....	24	Ward.....	16
Maverick.....	14	Washington.....	32
Medina.....	15	Webb.....	1
Menard.....	3	Wharton.....	34
Midland.....	6	Wheeler.....	7
Milam.....	19	Wichita.....	43
Mills.....	6	Wiltbarger.....	16
Mitchell.....	10	Willacy.....	26
Montague.....	16	Williamson.....	34
Montgomery.....	45	Wilson.....	16
Moore.....	15	Winkler.....	11
Morris.....	47	Wise.....	21
Motley.....	2	Wood.....	26
Nacogdoches.....	40	Yoakum.....	9
Navarro.....	29	Young.....	17
Newton.....	12	Zapata.....	5
Nolan.....	18	Zavala.....	9
Nueces.....	44	Utah:	
Ochiltree.....	11	Beaver.....	1
Oldham.....	2	Box Elder.....	5
Palo Pinto.....	31	Cache.....	36
Panola.....	18	Carbon.....	11
Parker.....	38	Daggett.....	1
Parmer.....	12	Duchesne.....	2
Pecos.....	3	Emery.....	1
Polk.....	13	Garfield.....	1
Potter.....	50	Grand.....	2
Presidio.....	1	Iron.....	4
Rains.....	18	Juab.....	1
Randall.....	50	Kane.....	1
Reagan.....	3	Millard.....	1
Real.....	3	Morgan.....	7
Red River.....	14	Piute.....	2
Reeves.....	6	Rich.....	2
Refugio.....	12	San Juan.....	1
Roberts.....	1	Sanpete.....	7
Robertson.....	16	Sevier.....	5
Rockwall.....	48	Summit.....	3
Runnels.....	11	Tooele.....	3
Rusk.....	36	Utah.....	3
Sabine.....	16	Utah.....	42
Sari Augustine.....	17	Wasatch.....	5
San Jacinto.....	11	Washington.....	6
San Saba.....	5	Wayne.....	1
Schleicher.....	2	Vermont:	
Scurry.....	17	Addison.....	31
Shackelford.....	4	Bennington.....	44
Shelby.....	25	Caledonia.....	37
Sherman.....	4	Essex.....	8
Smith.....	43	Franklin.....	47
Somervell.....	14	Grand Isle.....	43
Starr.....	15	Lamoille.....	28
Stephens.....	9	Orange.....	26
Sterling.....	1	Orleans.....	28
Stonewall.....	3	Windham.....	43
Sutton.....	2	Windsor.....	46
Swisher.....	12	Virginia:	
Taylor.....	10	Alleghany.....	28
Terrell.....	1	Amelia.....	21
Terry.....	16	Appomattox.....	28
Throckmorton.....	2	Augusta.....	45
Titus.....	40	Bath.....	16
Tom Green.....	5	Bedford.....	37

State/County	Population ¹	State/County	Population ¹
Bland.....	15	Pacific.....	17
Botetourt.....	33	Pend Oreille.....	4
Brunswick.....	28	San Juan.....	22
Buckingham.....	18	Skagit.....	30
Caroline.....	26	Skamania.....	3
Carroll.....	47	Stevens.....	7
Charles City.....	34	Wahkiakum.....	14
Charlotte.....	26	Walla Walla.....	33
Clarke.....	47	Whatcom.....	39
Craig.....	10	Whitman.....	18
Culpeper.....	47	Yakima.....	34
Cumberland.....	21	West Virginia:	
Dickenson.....	48	Barbour.....	41
Drumwidge.....	49	Braxton.....	25
Essex.....	28	Calhoun.....	25
Fauquier.....	40	Clay.....	27
Floyd.....	26	Doddridge.....	20
Fluvanna.....	26	Gilmer.....	23
Franklin.....	39	Grant.....	18
Giles.....	46	Greenbrier.....	31
Goochland.....	35	Hampshire.....	18
Grayson.....	34	Hardy.....	15
Greene.....	34	Jackson.....	45
Greensville.....	32	Lewis.....	46
Hallfax.....	38	Lincoln.....	43
Highland.....	6	Monroe.....	24
King and Queen.....	17	Morgan.....	37
King George.....	46	Nicholas.....	35
King William.....	27	Pendleton.....	10
Lee.....	46	Pocahontas.....	9
Louisa.....	27	Preston.....	39
Lunenburg.....	26	Randolph.....	24
Madison.....	26	Ritchie.....	22
Mecklenburg.....	48	Roane.....	29
Middlesex.....	48	Summers.....	38
Nelson.....	25	Tucker.....	18
New Kent.....	25	Tyler.....	39
Northumberland.....	49	Webster.....	18
Northway.....	46	Wirt.....	18
Orange.....	39	Wisconsin:	
Patrick.....	33	Adams.....	14
Powhatan.....	29	Ashland.....	16
Prince Edward.....	40	Barron.....	39
Rappahannock.....	19	Bayfield.....	8
Richmond.....	34	Buffalo.....	19
Rockbridge.....	28	Burnett.....	11
Scott.....	45	Chippewa.....	47
Shenandoah.....	45	Clark.....	25
Southampton.....	31	Crawford.....	27
Spotsylvania.....	40	Door.....	41
Surry.....	21	Douglas.....	34
Sussex.....	23	Dunn.....	34
Wythe.....	48	Florence.....	7
Washington:		Forest.....	8
Adams.....	6	Grant.....	42
Asotin.....	22	Jackson.....	5
Benton.....	39	Juneau.....	24
Chelan.....	14	Lafayette.....	22
Clallam.....	20	Langlade.....	22
Columbia.....	5	Green.....	46
Douglas.....	9	Green Lake.....	48
Ferry.....	2	Iowa.....	25
Franklin.....	21	Iron.....	9
Garfield.....	4	Lincoln.....	26
Grant.....	16	Marinette.....	26
Grays Harbor.....	31	Marquette.....	19
Jefferson.....	6	Menominee.....	7
Kittitas.....	11	Monroe.....	35
Klickitat.....	6	Oconto.....	26
Lewis.....	19	Oneida.....	22
Lincoln.....	4	Pepin.....	31
Mason.....	22	Pierce.....	45
Okanogan.....	5	Polk.....	29

State/County	Population ¹
Pierce.....	12
Richland.....	29
Rock.....	16
St. Croix.....	47
Sauk.....	46
Sawyer.....	8
Shawano.....	36
Taylor.....	17
Trempealeau.....	32
Vernon.....	31
Vilas.....	13
Washburn.....	13
Waushara.....	24
Wyoming.....	
Albany.....	6
Big Horn.....	3
Campbell.....	3
Carbon.....	2
Converse.....	1
Crook.....	2
Fremont.....	3
Goshen.....	5
Hot Springs.....	2
Johnson.....	1
Laramie.....	21
Lincoln.....	2
Natrona.....	10
Niobrara.....	1
Park.....	1
Platte.....	3
Shendan.....	7
Sublette.....	1
Sweetwater.....	2
Teton.....	1
Uinta.....	3
Washake.....	3
Weston.....	3

¹ County density (persons per square mile) excluding cities of 50,000 or more.

[39 FR 43285, Dec. 12, 1974, as amended at 40 FR 38156, Aug. 27, 1975]

APPENDIX D—SAMPLING PROCEDURES FOR FUEL VOLATILITY

1. Scope.

1.1 This method covers procedures for obtaining representative samples of gasoline for the purpose of testing for compliance with the Reid vapor pressure (RVP) standards set forth in § 80.27.

2. Summary of method.

2.1 It is necessary that the samples be truly representative of the gasoline in question. The precautions required to ensure the representative character of the samples are numerous and depend upon the tank, carrier, container or line from which the sample is being obtained, the type and cleanliness of the sample container, and the sampling procedure that is to be used. A summary of the sampling procedures and their application is presented in Table 1. Each procedure is suitable for sampling a material under definite storage, transportation, or contain-

er conditions. The basic principle of each procedure is to obtain a sample in such manner and from such locations in the tank or other container that the sample will be truly representative of the gasoline.

3. Description of terms.

3.1 "Average sample" is one that consists of proportionate parts from all sections of the container.

3.2 "All-levels sample" is one obtained by submerging a stoppered beaker or bottle to a point as near as possible to the draw-off level, then opening the sampler and raising it at a rate such that it is 70-85% full as it emerges from the liquid. An all-levels sample is not necessarily an average sample because the tank volume may not be proportional to the depth and because the operator may not be able to raise the sampler at the variable rate required for proportionate filling. The rate of filling is proportional to the square root of the depth of immersion.

3.3 "Running sample" is one obtained by lowering an unstoppered beaker or bottle from the top of the gasoline to the level of the bottom of the outlet connection or swing line, and returning it to the top of the gasoline at a uniform rate of speed such that the beaker or bottle is 70-85% full when withdrawn from the gasoline.

3.4 "Spot sample" is one obtained at some specific location in the tank by means of a thief bottle, or beaker.

3.5 "Top sample" is a spot sample obtained 6 inches (150 mm) below the top surface of the liquid (Figure 1).

3.6 "Upper sample" is a spot sample taken at the mid-point of the upper third of the tank contents (Figure 1).

3.7 "Middle sample" is a spot sample obtained from the middle of the tank contents (Figure 1).

3.8 "Lower sample" is a spot sample obtained at the level of the fixed tank outlet or the swing line outlet (Figure 1).

3.9 "Clearance sample" is a spot sample taken 4 inches (100 mm) below the level of the tank outlet (Figure 1).

3.10 "Bottom sample" is one obtained from the material on the bottom surface of the tank, container, or line at its lowest point.

3.11 "Drain sample" is one obtained from the draw-off or discharge valve. Occasionally, a drain sample may be the same as a bottom sample, as in the case of a tank car.

3.12 "Continuous sample" is one obtained from a pipeline in such manner as to give a representative average of a moving stream.

3.13 "Mixed sample" is one obtained after mixing or vigorously stirring the contents of the original container, and then pouring out or drawing off the quantity desired.

3.14 "Nozzle sample" is one obtained from a gasoline pump nozzle which dispenses gasoline from a storage tank at a retail outlet or a wholesale purchaser-consumer facility.

4. Sample containers.

4.1 Sample containers may be clear or brown glass bottles, or cans. The clear glass bottle is advantageous because it may be examined visually for cleanliness, and also allows visual inspection of the sample for free water or solid impurities. The brown glass bottle affords some protection from light. Cans with the seams soldered on the exterior surface with a flux of rosin in a suitable solvent are preferred because such a flux is easily removed with gasoline, whereas many others are very difficult to remove. If such cans are not available, other cans made with a welded construction that are not affected by, and that do not affect, the gasoline being sampled are acceptable.

4.2 Container closure. Cork or glass stoppers, or screw caps of plastic or metal, may be used for glass bottles; screw caps only shall be used for cans to provide a vapor-tight closure seal. Corks must be of good quality, clean and free from holes and loose bits of cork. Never use rubber stoppers. Contact of the sample with the cork may be prevented by wrapping tin or aluminum foil around the cork before forcing it into the bottle. Glass stoppers must be a perfect fit. Screw caps must be protected by a cork disk faced with tin or aluminum foil, or other material that will not affect petroleum or petroleum products.

4.3 Cleaning procedure. The method of cleaning all sample containers must be consistent with the residual materials in the container and must produce sample containers that are clean and free of water, dirt, lint, washing compounds, naphtha or other solvents, soldering fluxes, and acids, corrosion, rust, and oil. New sample containers should be inspected and cleaned if necessary. Dry either the container by passing a current of clean, warm air through the container or by allowing it to air dry in a clean area at room temperature. When dry, stopper or cap the container immediately.

5. Sampling apparatus.

5.1 Sampling apparatus is described in detail under each of the specific sampling procedures. Clean, dry, and free all sampling apparatus from any substance that might contaminate the material, using the procedure described in 4.3.

6. Time and place of sampling.

6.1 When loading or discharging gasoline, take samples from both shipping and receiving tanks, and from the pipeline if required.

6.2 Ship or barge tanks. Sample each product after the vessel is loaded or just before unloading.

6.3 Tank cars. Sample the product after the car is loaded or just before unloading.

NOTE: When taking samples from tanks suspected of containing flammable atmospheres, precautions should be taken to guard against ignitions due to static electricity. Metal or conductive objects, such as gage tapes, sample containers, and thermometers, should not be lowered into or suspended in a compartment or tank which is being filled or immediately after cessation of pumping. A waiting period of approximately one minute will generally permit a substantial relaxation of the electrostatic charge; under certain conditions a longer period may be deemed advisable.

7. Obtaining samples.

7.1 Directions for sampling cannot be made explicit enough to cover all cases. Extreme care and good judgment are necessary to ensure samples that represent the general character and average condition of the material. Clean hands are important. Clean gloves may be worn but only when absolutely necessary, such as in cold weather, or when handling materials at high temperature, or for reasons of safety. Select wiping cloths so that lint is not introduced, contaminating samples.

7.2 As many petroleum vapors are toxic and flammable, avoid breathing them or igniting them from an open flame or a spark produced by static. Follow all safety precautions specific to the material being sampled.

7.3 When sampling relatively volatile products (more than 2 pounds (0.14 kgf/cm²) RVP), the sampling apparatus shall be filled and allowed to drain before drawing the sample. If the sample is to be transferred to another container, this container shall also be rinsed with some of the volatile product and then drained. When the actual sample is emptied into this container, the sampling apparatus should be upended into the opening of the sample container and remain in this position until the contents have been transferred so that no unsaturated air will be entrained in the transfer of the sample.

8. Handling samples.

8.1 Volatile samples. It is necessary to protect all volatile samples of gasoline from evaporation. Transfer the product from the sampling apparatus to the sample container immediately. Keep the container closed except when the material is being transferred. After delivery to the laboratory, volatile samples should be cooled before the container is opened.

8.2 *Container outage.* Never completely fill a sample container, but allow adequate room for expansion, taking into consideration the temperature of the liquid at the time of filling and the probable maximum temperature to which the filled container may be subjected.

9. Shipping samples.

9.1 To prevent loss of liquid and vapors during shipment, and to protect against moisture and dust, cover the stoppers of glass bottles with plastic caps that have been swelled in water, wiped dry, placed over the tops of the stoppered bottles, and allowed to shrink tightly in place. The caps of metal containers must be screwed down tightly and checked for leakage. Postal and express office regulations applying to the shipment of flammable liquids must be observed.

10. Labeling sample containers.

10.1 Label the container immediately after a sample is obtained. Use waterproof and oilproof ink, or a pencil hard enough to dent the tag, since soft pencil and ordinary ink markings are subject to obliteration from moisture, oil smearing and handling. An indelible identification symbol, such as a bar code, may be used in lieu of a manually addressed label. The label shall reference the following information:

- 10.1.1 Date and time (the period elapsed during continuous sampling);
- 10.1.2 Name of the sample;
- 10.1.3 Name or number and owner of the vessel, car, or container;
- 10.1.4—Brand and grade of material; and
- 10.1.5—Reference symbol or identification number.

11. Sampling procedures.

11.1 The standard sampling procedures described in this method are summarized in Table 1. Alternative sampling procedures may be used if a mutually satisfactory agreement has been reached by the party(ies) involved and EPA and such agreement has been put in writing and signed by authorized officials.

11.2 *Bottle or beaker sampling.* The bottle or beaker sampling procedure is applicable for sampling liquids of 16 pounds (1.12 kgf/cm²) RVP or less in tank cars, tank trucks, shore tanks, ship tanks, and barge tanks.

11.2.1 *Apparatus.* A suitable sampling bottle or beaker as shown in Figure 2 is required. Recommended diameter of opening in the bottle or beaker is ¾ inch (19 mm).

11.2.2 Procedure.

11.2.2.1 *All-levels sample.* Lower the weighted, stoppered bottle or beaker as near as possible to the draw-off level, pull out the stopper with a sharp jerk of the cord or

chain and raise the bottle at a uniform rate so that it is 70-85% full as it emerges from the liquid.

11.2.2.2 *Running sample.* Lower the unstoppered bottle or beaker as near as possible to the level of the bottom of the outlet connection or swing line and then raise the bottle or beaker to the top of the gasoline at a uniform rate of speed such that it is 70-85% full when withdrawn from the gasoline.

11.2.2.3 *Upper, middle, and lower samples.* Lower the weighted, stoppered bottle to the proper depths (Figure 1) as follows:

- Upper sample..... middle of upper third of the tank contents
- Middle sample ... middle of the tank contents
- Lower sample..... level of the fixed tank outlet or the swing-line outlet

At the selected level pull out the stopper with a sharp jerk of the cord or chain and allow the bottle or beaker to fill completely, as evidenced by the cessation of air bubbles. When full, raise the bottle or beaker, pour off a small amount, and stopper immediately.

11.2.2.4 *Top sample.* Obtain this sample (Figure 1) in the same manner as specified in 11.2.2.3 but at six inches (150 mm) below the top surface of the tank contents.

11.2.2.5 *Handling.* Stopper and label bottle samples immediately after taking them, and deliver to the laboratory in the original sampling bottles.

11.3 *Tap sampling.* The tap sampling procedure is applicable for sampling liquids of twenty-six pounds (1.83 kgf/cm²) RVP or less in tanks which are equipped with suitable sampling taps or lines. This procedure is recommended for volatile stocks in tanks of the breather and balloon roof type, spheroids, etc. (Samples may be taken from the drain cocks of gage glasses, if the tank is not equipped with sampling taps.) The assembly for tap sampling is shown in Figure 3.

11.3.1 Apparatus.

11.3.1.1 *Tank taps.* The tank should be equipped with at least three sampling taps placed equidistant throughout the tank height and extending at least three feet (0.9 meter) inside the tank shell. A standard ¼ inch pipe with suitable valve is satisfactory.

11.3.1.2 *Tube.* A delivery tube that will not contaminate the product being sampled and long enough to reach to the bottom of the sample container is required to allow submerged filling.

11.3.1.3 *Sample containers.* Use clean, dry glass bottles of convenient size and

strength or metal containers to receive the samples.

11.3.2 Procedure. Before a sample is drawn, flush the tap (or gage glass drain cock) and line until they are purged completely. Connect the clean delivery tube to the tap. Draw upper, middle, or lower samples directly from the respective taps after the flushing operation. Stopper and label the sample container immediately after filling, and deliver it to the laboratory.

11.4 Continuous sampling. The continuous sampling procedure is applicable for sampling liquids of 16 pounds (1.12 kgf/cm²) RVP or less and semiliquids in pipelines, filling lines, and transfer lines. The continuous sampling may be done manually or by using automatic devices.

11.4.1 Apparatus.

11.4.1.1 Sampling probe. The function of the sampling probe is to withdraw from the flow stream a portion that will be representative of the entire stream. The apparatus assembly for continuous sampling is shown in Figure 4. Probe designs that are commonly used are as follows:

11.4.1.1.1 A tube extending to the center of the line and beveled at a 45 degree angle facing upstream (Figure 4(a)).

11.4.1.1.2 A long-radius forged elbow or pipe bend extending to the center line of the pipe and facing upstream. The end of the probe should be reamed to give a sharp entrance edge (Figure 4(b)).

11.4.1.1.3 A closed-end tube with a round orifice spaced near the closed end which should be positioned in such a way that the orifice is in the center of the pipeline and is facing the stream as shown in Figure 4(c)).

11.4.1.2 Probe location. Since the fluid to be sampled may not in all cases be homogeneous, the location, the position and the size of the sampling probe should be such as to minimize stratification or dropping out of heavier particles within the tube or the displacement of the product within the tube as a result of variation in gravity of the flowing stream. The sampling probe should be located preferably in a vertical run of pipe and as near as practicable to the point where the product passes to the receiver. The probe should always be in a horizontal position.

11.4.1.2.1 The sampling lines should be as short as practicable and should be cleared before any samples are taken.

11.4.1.2.2 Where adequate flowing velocity is not available, a suitable device for mixing the fluid flow to ensure a homogeneous mixture at all rates of flow and to eliminate stratification should be installed upstream of the sampling tap. Some effective devices for obtaining a homogeneous mixture are as follows: Reduction in pipe size; a series of baffles; orifice or perforated plate; and a combination of any of these methods.

11.4.1.2.3 The design or sizing of these devices is optional with the user, as long as the flow past the sampling point is homogeneous and stratification is eliminated.

11.4.1.3 To control the rate at which the sample is withdrawn, the probe or probes should be fitted with valves or plug cocks.

11.4.1.4 Automatic sampling devices that meet the standards set out in 11.4.1.5 may be used in obtaining samples of gasoline. The quality of sample collected must be of sufficient size for analysis, and its composition should be identical with the composition of the batch flowing in the line while the sample is being taken. An automatic sampler installation necessarily includes not only the automatic sampling device that extracts the samples from the line, but also a suitable probe, connecting lines, auxiliary equipment, and a container in which the sample is collected. Automatic samplers may be classified as follows:

11.4.1.4.1 Continuous sampler, time cycle (nonproportional) type. A sampler designed and operated in such a manner that it transfers equal increments of liquid from the pipeline to the sample container at a uniform rate of one or more increments per minute is a continuous sampler.

11.4.1.4.2 Continuous sampler, flow-responsive (proportional) type. A sampler that is designed and operated in such a manner that it will automatically adjust the quantity of sample in proportion to the rate of flow is a flow-responsive (proportional) sampler. Adjustment of the quantity of sample may be made either by varying the frequency of transferring equal increments of sample to the sample container, or by varying the volume of the increments while maintaining a constant frequency of transferring the increments to the sample container. The apparatus assembly for continuous sampling is shown in Figure 4.

11.4.1.4.3 Intermittent sampler. A sampler that is designed and operated in such a manner that it transfers equal increments of liquid from a pipeline to the sample container at a uniform rate of less than one increment per minute is an intermittent sampler.

11.4.1.5 Standards of installation. Automatic sampler installations should meet all safety requirements in the plant or area where used, and should comply with American National Standard Code for Pressure Piping, and other applicable codes (ANSI B31.1). The sampler should be so installed as to provide ample access space for inspection and maintenance.

11.4.1.5.1 Small lines connecting various elements of the installation should be so arranged that complete purging of the automatic sampler and of all lines can be accomplished effectively. All fluid remaining in the sampler and the lines from the preced-

ing sampling cycle should be purged immediately before the start of any given sampling operation.

11.4.1.5.2 In those cases where the sampler design is such that complete purging of the sampling lines and the sampler is not possible, a small pump should be installed in order to circulate a continuous stream from the sampling tube past or through the sampler and back into the line. The automatic sampler should then withdraw the sample from the sidestream through the shortest possible connection.

11.4.1.5.3 Under certain conditions, there may be a tendency for water and heavy particles to drop out in the discharge line from the sampling device and appear in the sample container during some subsequent sampling period. To circumvent this possibility, the discharge pipe from the sampling device should be free of pockets or enlarged pipe areas, and preferably should be pitched downward to the sample container.

11.4.1.5.4 To ensure clean, free-flowing lines, piping should be designed for periodic cleaning.

11.4.1.6 *Field calibration.* Composite samples obtained from the automatic sampler installation should be verified for quantity performance in a manner that meets with the approval of all parties concerned (including EPA), at least once a month and more often if conditions warrant. In the case of time-cycle samplers, deviations in quantity of the sample taken should not exceed $\pm$ five percent for any given setting. In the case of flow-responsive samplers, the deviation in quantity of sample taken per 1,000 barrels of flowing stream should not exceed $\pm$ five percent. For the purpose of field-calibrating an installation, the composite sample obtained from the automatic sampler under test should be verified for quality by comparing on the basis of physical and chemical properties, with either a properly secured continuous nonautomatic sample or tank sample. The tank sample should be taken under the following conditions:

11.4.1.6.1 The batch pumped during the test interval should be diverted into a clean tank and a sample taken within one hour after cessation of pumping.

11.4.1.6.2 If the sampling of the delivery tank is to be delayed beyond one hour, then the tank selected must be equipped with an adequate mixing means. For valid comparison, the sampling of the delivery tank must be completed within eight hours after cessation of pumping, even though the tank is equipped with a motor-driven mixer.

11.4.1.6.3 When making a normal full-tank delivery from a tank, a properly secured sample may be used to check the results of the sampler if the parties (including EPA) mutually agree to this procedure.

11.4.1.7 *Receiver.* The receiver must be a clean, dry container of convenient size to receive the sample. All connections from the sample probe to the sample container must be free of leaks. Two types of containers may be used, depending upon service requirements.

11.4.1.7.1 *Atmospheric container.* The atmospheric container shall be constructed in such a way that it retards evaporation loss and protects the sample from extraneous material such as rain, snow, dust, and trash. The construction should allow cleaning, interior inspection, and complete mixing of the sample prior to removal. The container should be provided with a suitable vent.

11.4.1.7.2 *Closed container.* The closed container shall be constructed in such a manner that it prevents evaporation loss. The construction must allow cleaning, interior inspection and complete mixing of the sample prior to removal. The container should be equipped with a pressure-relief valve.

11.4.2 Procedure.

11.4.2.1 *Nonautomatic sample.* Adjust the valve or plug cock from the sampling probe so that a steady stream is drawn from the probe. Whenever possible, the rate of sample withdrawal should be such that the velocity of liquid flowing through the probe is approximately equal to the average linear velocity of the stream flowing through the pipeline. Measure and record the rate of sample withdrawal as gallons per hour. Divert the sample stream to the sampling container continuously or intermittently to provide a quantity of sample that will be of sufficient size for analysis.

11.4.2.2 *Automatic sampling.* Purge the sampler and the sampling lines immediately before the start of a sampling operation. If the sample design is such that complete purging is not possible, circulate a continuous stream from the probe past or through the sampler and back into the line. Withdraw the sample from the side stream through the automatic sampler using the shortest possible connections. Adjust the sampler to deliver not less than one and not more than 40 gallons (151 liters) of sample during the desired sampling period. For time-cycle samplers, record the rate at which sample increments were taken per minute. For flow-responsive samplers, record the proportion of sample to total stream. Label the samples and deliver them to the laboratory in the containers in which they were collected.

11.5 *Nozzle sampling.* The nozzle sampling procedure is applicable for sampling gasoline from a retail outlet or wholesale purchaser-consumer facility storage tank.

11.5.1 *Apparatus.* Sample containers conforming with § 4.1 should be used. A spacer, if appropriate (figure 6), and a nozzle exten-

sion device similar to that shown in Figures 7 or 7a shall be used when nozzle sampling. The nozzle extension device does not need to be identical to that shown in Figures 7 or 7a but it should be a device that will bottom fill the container.

11.5.2 Procedure. Immediately after gasoline has been delivered from the pump and the pump has been reset, deliver a small amount of product through the nozzle extension into the sample container using a spacer (Figure 6) if needed, on the pump nozzle (vapor recovery type). Rinse the sample container and dump the product into waste container. Insert nozzle extension (Figure 7 or 7a) into sample container and insert pump nozzle into extension with slot over the air bleed hole (when using Figure 7). Fill sample container slowly through nozzle extension to 70-80 percent full (Figure 8). Remove nozzle extension. Cap sample container at once. Check for leaks. Discard sample container and resample if leak occurs. If sample container is leak tight, label the container and deliver it to the laboratory.

12. Special Precautions and Instructions.

12.1 Precautions. Vapor pressures are extremely sensitive to evaporation losses and to slight changes in composition. When obtaining, storing, or handling samples, observe the necessary precautions to ensure samples representative of the product and satisfactory for RVP tests. Official samples should be taken by, or under the immediate supervision of, a person of judgment, skill, and sampling experience. Never prepare composite samples for this test. Make certain that containers which are to be shipped by common carrier conform to applicable Interstate Commerce Commission, state, and local regulations. When flushing or purging lines or containers, observe the pertinent regulations and precautions against fire, explosion, and other hazards.

12.2 Sample containers. Use containers of not less than one quart (0.9 liter) nor more than two gallons (7.6 liters) capacity, of sufficient strength to withstand the pressures to which they may be subjected, and of a type that will permit replacement of the cap or stopper with suitable connections for transferring the sample to the gasoline chamber of the vapor pressure apparatus. Open-type containers have a single opening which permits sampling by immersion. Closed-type containers have two openings, one in each end (or the equivalent thereof), fitted with valves suitable for sampling by water displacement or by purging.

12.3 Transfer connections. The transfer connection for the open-type container consists of an air tube and a liquid delivery tube assembled in a cap or stopper. The air tube extends to the bottom of the container. One end of the liquid delivery tube is flush with

the inside face of the cap or stopper and the tube is long enough to reach the bottom of the gasoline chamber while the sample is being transferred to the chamber. The transfer connection for the closed-type container consists of a single tube with a connection suitable for attaching it to one of the openings of the sample container. The tube is long enough to reach the bottom of the gasoline chamber while the sample is being transferred.

12.4 Sampling open tanks. Use clean containers of the open type when sampling open tanks and tank cars. An all-level sample obtained by the bottle procedure described in 11.2 is recommended. Before taking the sample, flush the container by immersing it in the product to be sampled. Then obtain the sample immediately. Pour off enough so that the container will be 70-80 percent full and close it promptly. Label the container and deliver it to the laboratory.

12.5 Sampling closed tanks. Containers of the closed type may be used to obtain samples from closed or pressure tanks. Obtain the sample using the water displacement procedure described in 12.6 or the purging procedure described in 12.7. The water displacement procedure is preferable because the flow of product involved in the purging procedure may be hazardous.

12.6 Purging procedure. Connect the inlet valve of the closed-type container to the tank sampling tap or valve. Throttle the outlet valve of the container so that the pressure in it will be approximately equal to that in the container being sampled. Allow a volume of product equal to at least twice that of the container to flow through the sampling system. Then close all valves, the outlet valve first, the inlet valve of the container second, and the tank sampling valve last, and disconnect the container immediately. Withdraw enough of the contents so that the sample container will be 70-80 percent full. If the vapor pressure of the product is not high enough to force liquid from the container, open both the upper and lower valves slightly to remove the excess. Promptly seal and label the container, and deliver it to the laboratory.

TABLE 1—SUMMARY OF GASOLINE SAMPLING PROCEDURES AND APPLICABILITY

Type of container	Procedure	Paragraph
Storage tanks, ship and barge tanks, tank cars, tank trucks.	Bottle sampling	11.2
Storage tanks with taps	Tap sampling	11.3
Pipes and lines	Continuous line sampling	11.4

TABLE 1—SUMMARY OF GASOLINE SAMPLING
PROCEDURES AND APPLICABILITY—Continued

Type of container	Procedure	Paragraph
Retail outlet and whole-sale purchaser-consumer facility storage tanks.	Nozzle sampling.	11.5

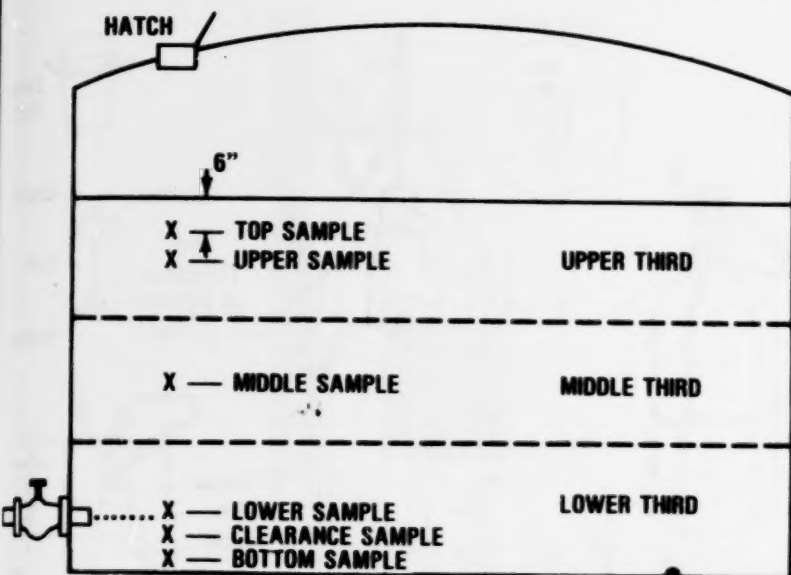
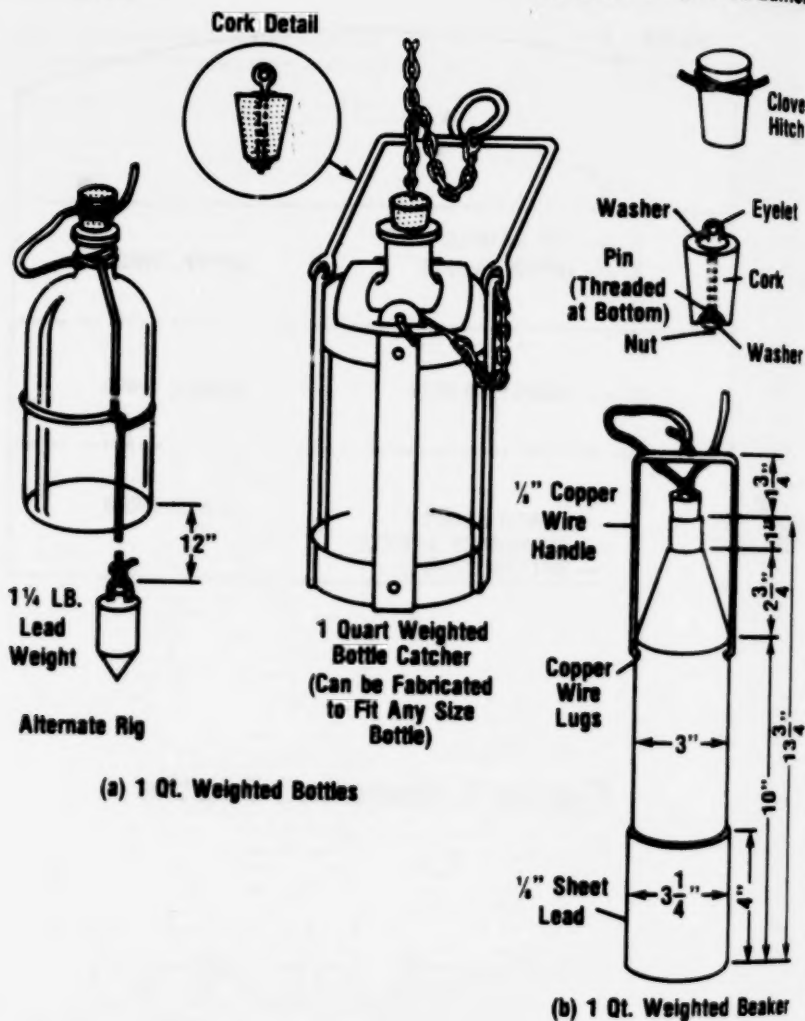


Figure 1. Sampling Depths



METRIC EQUIVALENTS

in.	1/8	1	1 1/4	2	3/4	4	10	12	13 1/2
mm	3	25	45	70	83	102	250	300	350

Figure 2. Assembly for Bottle Sampling

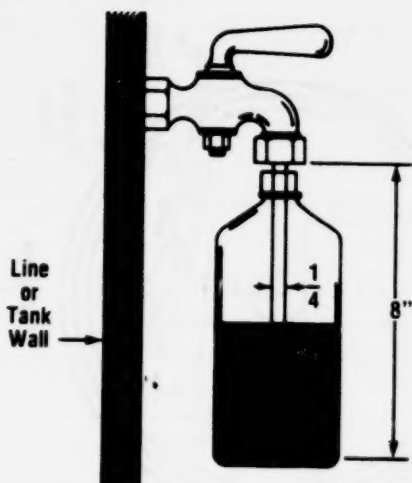
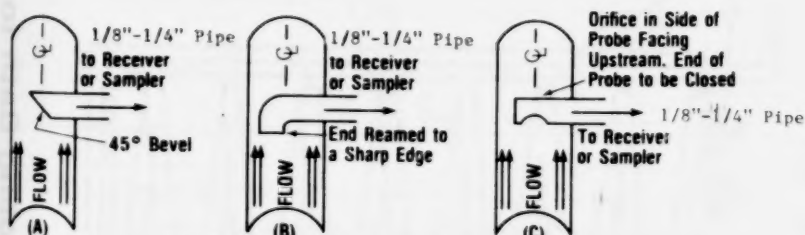


Figure 3. Assembly for Tap Sampling



Note: Probe may be fitted with valves or plug cocks.
Probe should be disposed horizontally.

PROBES FOR CONTINUOUS SAMPLING



TYPICAL ASSEMBLY FOR LINE SAMPLING

Figure 4. Probes for Continuous Sampling

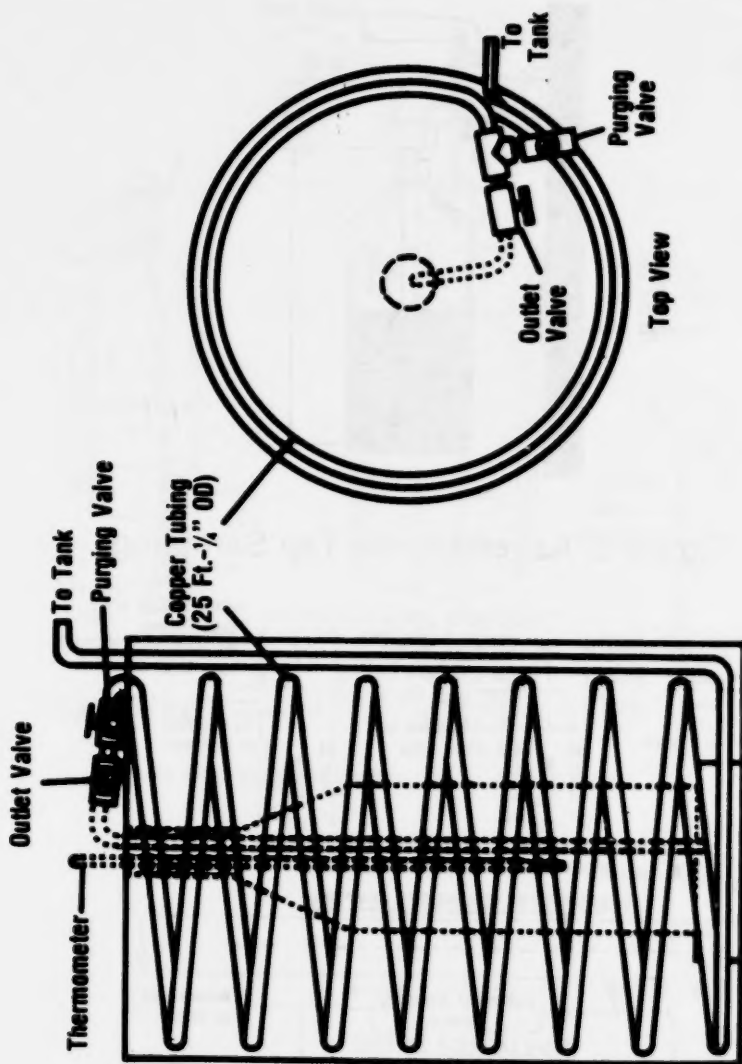
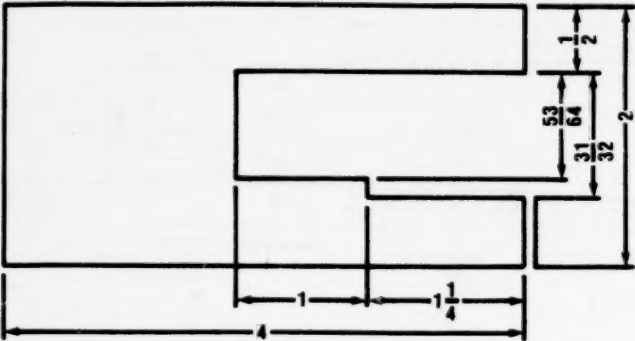
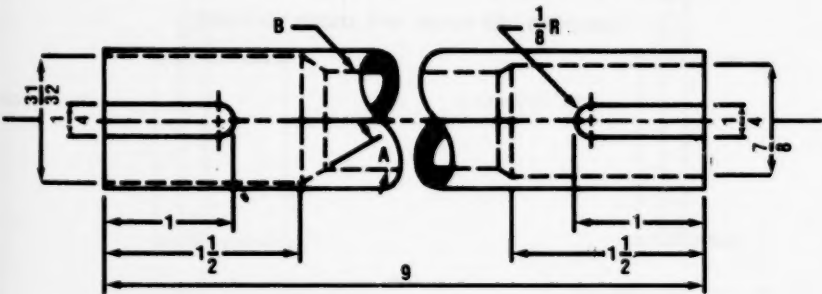


Figure 5. Cooling Bath for Reid Vapor Pressure Sampling



Make from 1/4 inch flat steel
All dimensions in inches
Break all edges and corners

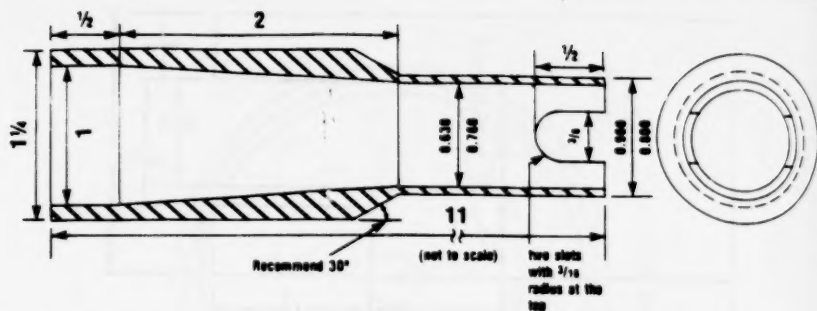
Figure 6. Spacer for Nozzle Sampling



Use 1/2 in. Schedule 80 Black Iron Pipe
All dimensions in inches
All tolerances $\pm 1/32$

A—Recommend 30°
B—Inside Diameter Schedule 80 Black Iron Pipe

Figure 7. Nozzle Extension for Nozzle Sampling



All Dimensions in inches (full scale except as noted)
 All decimal dimensions represent minimum and maximum
 Tolerance for all other dimensions is $\pm \frac{1}{32}$ "
 Made of non-ferrous material, unaffected by gasoline

Figure 7a. Nozzle Extension for Nozzle Sampling
 (Compatible with narrow neck sample containers)

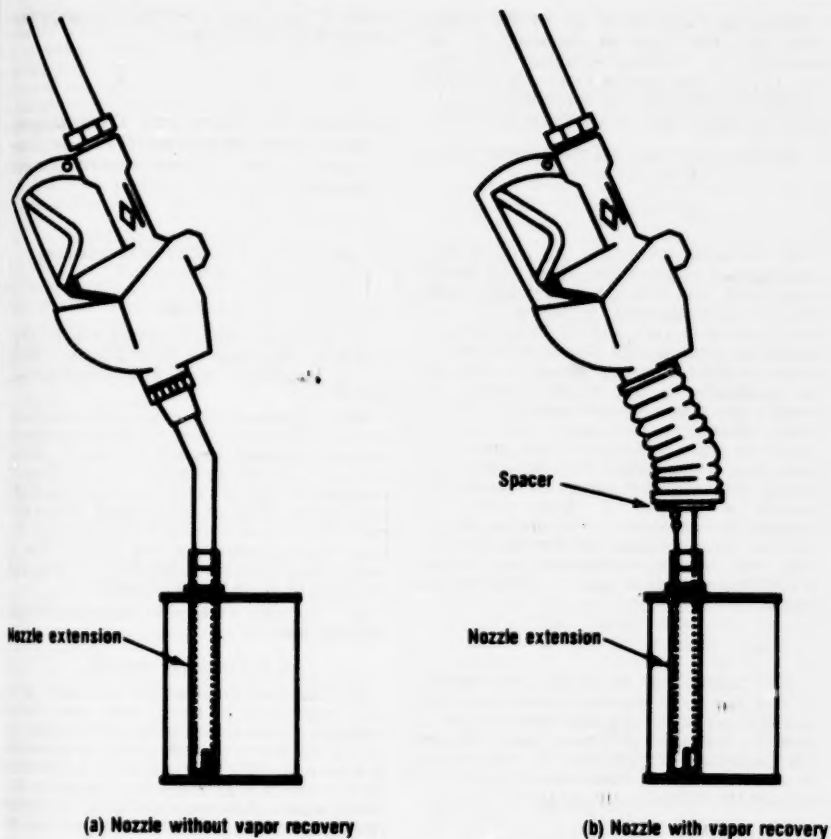


Figure 8. Assembly for Nozzle Sampling

[54 FR 11886, Mar. 22, 1989; 54 FR 27017, June 27, 1989, as amended at 55 FR 25835, June 25, 1990]

EFFECTIVE DATE NOTE: At 55 FR 25835, June 25, 1990, Part 80, Appendix D was amended by revising sections 4.3, 10.1, 11.5.1, 11.5.2 and adding Figure 7a effective August 24, 1990. For the convenience of the user the superseded text appears as follows.

APPENDIX D—SAMPLING PROCEDURES FOR FUEL VOLATILITY

4.3 Cleaning procedure. All sample containers must be absolutely clean and free of water, dirt, lint, washing compounds, naphtha, or other solvents, soldering fluxes or acids, corrosion, rust, and oil. Before using a container, rinse it with Stoddard solvent or other naphtha of similar volatility. (It may be necessary to use sludge solvents to remove all traces of sediment and sludge from containers previously used.) Then wash the container with strong soap solution, rinse it thoroughly with tap water, and finally with distilled water. Dry either by passing a current of clean, warm air through the container or by placing it in a hot dust-free cabinet at 104 degrees Fahrenheit (40 degrees centigrade) or higher. When dry, stopper or cap the container immediately.

10.1 Label the container immediately after a sample is obtained. Use waterproof and oilproof ink or a pencil hard enough to dent the tag, since soft pencil and ordinary ink markings are subject to obliteration from moisture, oil smearing and handling. Include the following information:

11.5.1 *Apparatus.* Sample containers conforming with 4.1 should be used. A spacer, if appropriate, and a nozzle extension as shown in Figures 6 and 7 shall be used when nozzle sampling.

11.5.2 *Procedure.* Immediately after gasoline has been delivered from the pump and the pump has been reset, deliver a small amount of product into the sample container, using a spacer (Figure 6), if needed, on the pump nozzle (vapor recovery type). Rinse sample container and dump product into waste container. Insert nozzle extension (Figure 7) into sample container and insert pump nozzle into extension with slot over air bleed hole. Fill sample container slowly through nozzle extension to 70-80 percent full (Figure 8). Remove nozzle extension. Cap sample container at once. Check for leaks. Discard sample container and resample if leak occurs. If sample con-

tainer is leak tight, label the container and deliver it to the laboratory.

APPENDIX E—TESTS FOR DETERMINING REID VAPOR PRESSURE (RVP) OF GASOLINE AND GASOLINE-OXYGENATE BLENDS

METHOD 1—DRY RVP MEASUREMENT METHOD.

1. Scope.

1.1 This test method covers the determination of the absolute vapor pressure (Note 1) of gasolines and gasoline-oxygenate blends.

NOTE 1: Because the external atmospheric pressure is counteracted by the atmospheric pressure initially present in the air chamber, the "vapor pressure" is an absolute pressure at 100 °F (37.8 °C) in pounds-force per square inch or kilopascals (kPa=kN/m²). This vapor pressure differs from the true vapor pressure of the sample due to some small sample vaporization and the presence of air in the confined space.

1.2 The values stated in inch-pound units are standard.

2. Summary of method.

2.1 The fuel chamber of the vapor pressure apparatus is filled with the chilled sample and connected to the air chamber at 100 °F (37.8 °C). The apparatus is immersed in a bath at 100 °F and is shaken periodically until a constant pressure is observed on the gauge attached to the apparatus. The gauge reading, suitably corrected, is reported as the vapor pressure.

3. Significance and use.

3.1 Test method ASTM D-323 cannot be used to determine the vapor pressure of gasoline-oxygenate blends which contain water-extractable oxygenates because the fuel sample comes into contact with water. This test method is a modification of Test Method ASTM D-323 where contact with water has been eliminated.

4. Apparatus.

4.1 The construction of the required apparatus is described in Annex A1.1 of this Appendix.

5. Reagents.

5.1 Purity of reagents. Use reagent grade chemicals in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee

Analytical Reagents of the American Chemical Society where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

5.2 Acetone (Danger—Extremely flammable. See Annex A3).

5.3 Naphtha (Danger—Extremely flammable. See Annex A2).

6. Handling of samples.

6.1 The extreme sensitivity of vapor pressure measurements to losses through evaporation and the resulting changes in composition is such as to require the utmost precaution and the most meticulous care in the handling of samples. The provisions of this section apply to all samples for vapor pressure determinations.

6.2 Sample in accordance with 40 CFR Part 80, Appendix D.

6.3 Sample container size. The size of the sample container from which the vapor pressure sample is taken is 1 quart (1 liter). It will be 70 to 85% filled with the sample.

6.4 Precautions.

6.4.1 Determine vapor pressure as the first test run on a sample. Do not withdraw more than one sample from the sample container for this test.

6.4.2 Protect samples from excessive heat prior to testing.

6.4.3 Do not test samples in leaky containers. Discard them and obtain new samples.

6.4.4 Discard samples that have separated into two phases and obtain new samples (see Note 4).

6.4.5 Sample handling temperature. In all cases, cool the sample container and contents to 32 to 34 °F (0 to 1 °C) before the container is opened. To ensure sufficient time to reach this temperature, directly measure the temperature of a similar liquid at a similar initial temperature in a like container placed in the cooling bath at the same time as the sample.

7. Preparation for test.

7.1 Verification of sample container filling. With the sample at a temperature of 32 to 34 °F (0 to 1 °C), take the container from the cooling bath, wipe dry with an absorbent material, unseal it, and examine its filling. The sample content, as determined by use of a suitable gauge, must be equal to 70 to 80% of the container capacity.

7.1.1 Discard the sample if its volume is less than 70% of the container capacity.

7.1.2 If the container is more than 80% full, pour out enough sample to bring the container contents within the 70 to 80% range. Under no circumstance may any

sample poured out be returned to the container.

7.2 Air saturation of sample in sample container.

7.2.1 With the sample at a temperature of 32 to 34 °F (0 to 1 °C) take the container from the cooling bath, wipe it dry with an absorbent material, unseal it momentarily, taking care to prevent water entry, reseal it, and shake it vigorously. Return it to the bath for a minimum of 2 minutes.

7.2.2 Repeat 7.2.1 twice more. Return the sample to the bath and keep there until the beginning of the procedure (8.1).

7.3 Preparation of fuel chamber. Observe the apparatus preparation procedure of 8.5, then store the stoppered fuel chamber and the sample transfer connection in a refrigerator or ice-water bath for a sufficient time to allow the chamber and the connection to reach a temperature of 32 to 34 °F (0 to 1 °C). If an ice-water bath is used, keep the chamber upright, corked, and not immersed over the top of the coupling threads. The transfer connection is inserted into a plastic bag to keep it completely dry during cooling.

7.4 Preparation of air chamber. Observe the apparatus preparation procedure of 8.5. Connect the gage to the air chamber and close the lower opening securely with a dry No. 6½ rubber stopper. Make sure the stopper is inserted far enough to securely close the vent hole in the air chamber connection. Immerse the air chamber to at least 1 inch (25 mm.) above its top in the water bath maintained at 100 ± 0.2 °F (37.8 ± 0.1 °C) for not less than 20 minutes. Do not remove the air chamber from the water bath until the fuel chamber has been filled with the sample as described in 8.1.

8. Procedure

8.1 Sample transfer. With everything in readiness, remove the chilled sample container from the bath, dry it with absorbent material, uncap it, dry and insert the chilled transfer apparatus (see Fig. 1.1). After drying with absorbent material, quickly place the chilled fuel chamber, in an inverted position, over the sample delivery tube of the transfer apparatus. Invert the entire system rapidly so that the fuel chamber is upright, with the end of the delivery tube touching the bottom of the fuel chamber. Fill the fuel chamber to overflowing. Withdraw the delivery tube from the fuel chamber while allowing the sample to continue flowing up to the moment of complete withdrawal.

8.1.1 Caution. Make provision for suitable collection and disposal of the overflowing fuel to avoid fire hazard.

8.2 Assembly of apparatus. Immediately remove the air chamber from the water bath and immediately dry the exterior of

the chamber with absorbent material, giving particular care to the connection between the air chamber and the fuel chamber. Remove the stopper after drying and immediately couple the two chambers. Not more than 10 seconds shall be consumed in coupling the two chambers.

NOTE 2: When the air chamber is removed from the water bath, is dried, and the stopper is removed, connect it to the fuel chamber without undue movements through the air, which could promote exchange of room temperature air with the 100 °F (37.8 °C) air in the chamber.

8.3 Introduction of apparatus into bath. Turn the assembled vapor pressure apparatus upside down to allow the sample in the fuel chamber to run into the air chamber. With apparatus still inverted, shake it vigorously eight times in a direction parallel to the length of the apparatus. With the gage end up, immerse the assembled apparatus in the bath, maintained at $100 \pm 0.2^\circ\text{F}$ ($37.8 \pm 0.1^\circ\text{C}$), in an inclined position so that the connection of the fuel and air chambers is below the water level and may be carefully examined for leaks. If no leaks are observed, further immerse the apparatus to at least 1 inch (25 mm.) above the top of the air chamber. Observe the apparatus for leakage throughout the test. Discard the test at any time a leak is detected.

NOTE 3: Liquid leaks are more difficult to detect than vapor leaks; because the coupling between the chambers is normally in the liquid section of the apparatus, give the coupling particular attention.

NOTE 4: After the apparatus has been immersed in the bath, check the remaining sample for phase separation. If the sample is contained in a glass container, this observation can be made prior to sample transfer (8.1). If the sample is contained in a non-transparent container, shake the sample vigorously for 5 seconds and then immediately pour a portion of the remaining sample into a clear glass container. Immediately after shaking this sample again for 5 seconds, observe the sample for phase separation. If this sample is not clear and bright, record the sample as cloudy. If this sample has more than one phase, record the sample as having phase separation.

8.4 Measurement of vapor pressure. After the assembled vapor pressure apparatus has been immersed in the bath for at least 5 minutes, tap the pressure gage lightly and observe the reading. Withdraw the apparatus from the bath and repeat 8.3. At intervals of not less than 2 minutes, perform 8.3 until a total of not less than five shakings and gage readings have been made; continue thereafter, if necessary, until the last two consecutive gage readings are constant, indicating equilibrium attainment. These operations normally require 20 to 30 minutes. Read the final gage pressure to the

nearest 0.05 psi (0.25 kPa) for gages with intermediate graduations of 0.1 psi (0.5 kPa) or less and to the nearest 0.1 psi for gages with graduations of 0.2 to 0.5 psi (1.0 to 2.5 kPa), and record the value as the "uncorrected vapor pressure" of the sample. Without undue delay remove the pressure gage and, without attempting to remove any liquid which may be trapped in the gage, check its reading against that of the manometer while both are subjected to a common steady pressure which is no more than 0.2 psi (1.0 kPa) different from the recorded "uncorrected vapor pressure". If a difference is observed between the gage and manometer readings, the difference shall be added to or subtracted from the "uncorrected vapor pressure" recorded for the sample being tested, and the resulting value shall be recorded as the vapor pressure of the sample.

NOTE 5: Cooling the assembly prior to disconnecting the gage will facilitate disassembly and reduce the amount of hydrocarbon vapors released in the room.

NOTE 6: Verification of Sample Integrity. Disconnect the air chamber from the fuel chamber. Drain the sample from the air and fuel chambers as completely as possible into a dry 8-ounce clear glass bottle. Seal the bottle and shake it vigorously for 5 seconds. If the sample is clear and bright and free of a second phase, note this observation and record that the test is valid. If the sample is not clear and bright and free of a second phase, immerse the bottle in the 100 °F (37.8 °C) water bath up to about 1 inch (25 mm.) above the top of the sample level for 15 minutes in order to heat the sample to the test temperature. Remove the sample from the water bath and immediately shake it vigorously for 5 seconds and observe the sample. If the sample is not clear and bright and free of a second phase, note this observation and record that the test is not valid because of phase separation. A fuel that is not clear and bright and free of a second phase at this point of the test indicates that the fuel was contacted with sufficient water to exceed the water tolerance of the fuel during the test procedure. Water can most likely get into the test chambers during preparation of the fuel and air chambers (7.3 and 7.4) or assembly of the air and fuel chambers (8.2), or both, especially if water baths are used for these procedures.

8.5 Preparation of apparatus for next test. Either of the cleaning procedures listed in sections 8.5.1 and 8.5.2 may be used to prepare the apparatus for the next test.

8.5.1 Thoroughly purge the air chamber of residual sample by filling it with warm water above 90 degrees F (32 degrees C) and allowing it to drain (Note 7). Repeat this purging at least five times. After disconnecting the pressure gage from its manifold con-

nection with the manometer, remove trapped fluid in the Bourdon tube of the gage by repeated centrifugal thrusts. This may be accomplished in the following manner: hold the gage between the palms of the hands with the right hand on the face side and the threaded connection of the gage forward. Extend the arms forward and upward at an angle of 45 degrees with the coupling of the gage pointing in the same direction. Swing the arms downward through an arc of about 135 degrees so that the centrifugal force aids gravity in removing the trapped liquid. Repeat this operation three times to expel all liquid. Purge the pressure gage by directing a small jet of air into its Bourdon tube for at least 5 minutes. Rinse both chambers and the transfer connection several times with hot water, then several times with acetone, they dry by blowing dried and filtered compressed air or pulling a vacuum. Stopper the fuel chamber and place it in the refrigerator or ice-water bath for the next test.

NOTE 7: If the purging of the air chamber is done in a bath, be sure to avoid small and unnoticeable films of floating sample by keeping the bottom and top openings of the chamber closed as they pass through the water surface.

8.5.2 Drain residual sample from the air and gasoline chambers. Rinse the chambers and the transfer connections in a manner such that all the inner surfaces are wetted with commercial grade n-pentane and drain into an appropriate container. Dry the interior surfaces with dried and filtered compressed air or by pulling a vacuum through them. After disconnecting the pressure gage from its manifold connection with the manometer, remove trapped fluid in the Bourdon tube of the gage by repeated centrifugal thrusts. This may be accomplished in the following manner: Hold the gage between the palms of the hands with the right hand on the face side and the threaded connection of the gage forward. Extend the arms forward and upward at an angle of 45 degrees with the coupling of the gage pointing in the same direction. Swing the arms downward through an arc of about 135 degrees so that the centrifugal force aids gravity in the removing of the trapped liquid. Repeat this operation three times to expel all liquid. Purge the pressure gage by directing a small jet of air into its Bourdon tube for at least 5 minutes. Stopper the fuel chamber and place it in the refrigerator or ice-water bath for the next test.

9. Precautions

9.1 Gross errors can be obtained in vapor pressure measurements if the prescribed

procedure is not followed carefully. The following list emphasizes the importance of strict adherence to the precautions given in the procedure.

9.1.1 Checking the pressure gage. Check all gages against a manometer after each test in order to ensure high precision of results (8.4). Read all gages while the gages are in a vertical position and after tapping them lightly.

9.1.2 Shake the container vigorously to ensure equilibrium of the sample with the air in the container (7.2).

9.1.3 Checking for leaks. Check the apparatus before and during each test for both liquid and vapor leaks (Annex A1.1.6 to this Appendix and Note 3).

9.1.4 Check O-rings before each test for cracking and clean if necessary.

9.1.5 Sampling. Because initial sampling and the handling of samples will greatly affect the final results, employ the utmost precaution and the most meticulous care to avoid losses through evaporation and even slight changes in composition (6.5 and 8.1). In no case shall any part of the apparatus itself be used as the sample container previous to actually conducting the test.

9.1.6 Purging the apparatus. Thoroughly purge the pressure gage, the fuel chamber and the air chamber to be sure they are free of residual sample. (This is most conveniently done at the end of the previous test. See 8.5). It is important to remove all water from the apparatus before cooling the gasoline chambers and heating the air chamber. In high-humidity conditions be alert for and avoid condensation on the transfer connection and interior walls of the apparatus.

9.1.7 Coupling the apparatus. Carefully observe the requirements of 8.2.

9.1.8 Shaking the apparatus. Shake the apparatus "vigorously" as directed in 8.3 in order to ensure equilibrium.

10. Report.

10.1 Reporting results. Report to the nearest 0.05 psi (0.25 kPa) or 0.1 psi (0.5 kPa) the gage result observed in 8.4, after correcting for any difference between the gage and manometer, as the "vapor pressure" in pounds-force per square inch (or kilopascals) without reference to temperature.

11. Precision and Accuracy

11.1 Precision. The precision of this test method has not been determined.

11.2 Accuracy. The accuracy of this test method has not been determined.

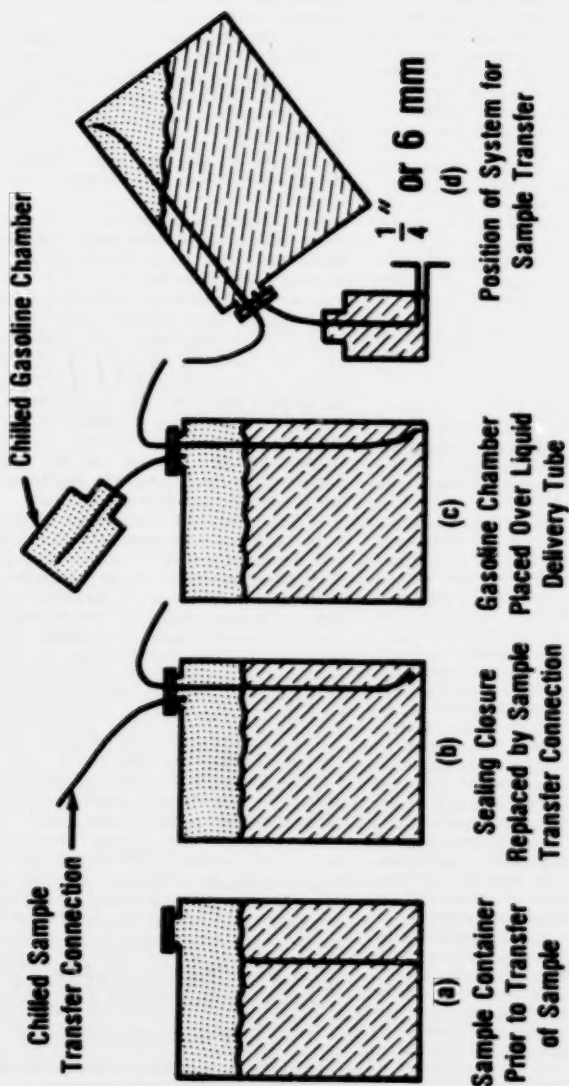


Figure 1.1 Simplified Sketches Outlining Method of Transferring Samples of Gasoline Chamber from Open-Type Containers

ANNEX A1. APPARATUS FOR REID VAPOR PRESSURE TEST

A1.1 Reid Vapor Pressure Bomb, consisting of two chambers, an air chamber (upper section) and a gasoline chamber (lower section) shall conform to the following requirements:

NOTE A1.1: Caution—To maintain the correct volume ratio between the air chamber and the gasoline chamber, the units shall not be interchanged without recalibrating to ascertain that the volume ratio is within satisfactory limits.

A1.1.1 Air Chamber—The upper section or air chamber, as shown in Fig. A1.1, shall be a cylindrical vessel $2 \pm \frac{1}{8}$ inches (51 ± 3 mm.) in diameter and $10 \pm \frac{1}{8}$ inches (254 ± 3 mm.) in length, inside dimensions, with the inner surfaces of the ends slightly sloped to provide complete drainage from either end when held in a vertical position. On one end of the air chamber, a suitable gage coupling with an internal diameter not less than $\frac{3}{16}$ inch shall be provided to receive the $\frac{1}{4}$ inch gage connection. In the other end of the air chamber an opening approximately $\frac{1}{2}$ inch in diameter shall be provided for coupling with the gasoline chamber. Care shall be taken that the connections to the end openings do not prevent the chamber from draining completely.

A1.1.2 Gasoline Chamber (One-Opening)—The lower section or gasoline chamber, as shown in Fig. A1.1, shall be a cylindrical vessel of the same inside diameter as the air chamber and of such volume that the ratio of the volume of the air chamber to the volume of gasoline chamber shall be between the limits of 3.8 and 4.2. In one end of the gasoline chamber an opening approximately $\frac{1}{2}$ inch in diameter shall be provided for coupling with the air chamber. The inner surface of the end containing the coupling member shall be sloped to provide complete drainage when inverted. The other end of the gasoline chamber shall be completely closed.

A1.1.3 Gasoline Chamber (Two-Opening)—For sampling from closed vessels, the lower section or gasoline chamber, as shown in Fig. A1.1 shall be essentially the same as the gasoline chamber described in A1.1.2, except that a $\frac{1}{4}$ inch valve shall be attached near the bottom of the gasoline chamber and a $\frac{1}{2}$ inch straight-through, full-opening valve shall be introduced in the coupling between the chambers. The volume of the gasoline chamber, including only the capacity enclosed by the valves, shall fulfill the volume ratio requirements as set forth in A1.1.2.

NOTE A1.2: In determining capacities for the two-opening gasoline chamber (Fig. A1.1), the capacity of the gasoline chamber shall be considered as that below the $\frac{1}{2}$ inch valve closure. The volume above the $\frac{1}{2}$ inch

valve closure, including the portion of the coupling permanently attached to the gasoline chamber, shall be considered as a part of the air chamber capacity.

A1.1.4 Method of Coupling Air and Gasoline Chambers—Any method of coupling the air and gasoline chambers may be employed, provided that no gasoline is lost during the coupling operation, that no compression effect is caused by the act of coupling, and that the assembly is free of leaks under the conditions of the tests. To avoid displacement of gasoline during assembly, it is desirable that the male fitting of a suitable coupling be on the gasoline chamber. To avoid compression of air during the assembly of a suitable screw coupling, a vent hole may be used to ensure atmospheric pressure in the air chamber at the instant of sealing.

NOTE A1.3: Caution—Some commercially available equipment does not make adequate provision for avoiding air compression effects. Before employing any apparatus, it shall be established that the act of coupling does not compress the air in the air chamber. This may be accomplished by tightly stoppering the gasoline chamber opening and assembling the apparatus in the normal manner, utilizing the 0 to 5-psi (0 to 35-kPa) gage. Any observable pressure increase on the gage is an indication that the apparatus does not adequately meet the specifications of the method. If this problem is encountered, the manufacturer should be consulted for remedy.

A1.1.5 Volumetric Capacity of Air and Gasoline Chambers—In order to ascertain if the volume ratio of the chambers is between the specified limits of 3.8 to 4.2, measure a quantity of water greater than will be needed to fill the gasoline and air chambers. The gasoline chamber shall be completely filled with water, and the difference between the original volume and the remaining volume is the volume of the gasoline chamber. Then, after connecting the gasoline and air chambers, the air chamber shall be filled to the seat of the gage connection with more of the measured water, and the difference in volumes shall be the volume of the air chamber.

A1.1.6 Checking for Freedom of Leak—Before placing new apparatus in service and as often as necessary thereafter, the assembled vapor pressure apparatus shall be checked for freedom of leaks by filling with air to 100-psi (700-kPa) gage pressure and completely immersing in a water bath. Only apparatus which stand this test without leaking shall be used.

A1.2 Pressure Gage—The pressure gage shall be a Bourdon-type spring gage of test gage quality $4 \frac{1}{2}$ to $5 \frac{1}{2}$ inches (100 to 150 mm) in diameter provided with a nominal $\frac{1}{4}$ inch male thread connection with a passage-

way not less than $\frac{3}{16}$ inch in diameter from the Bourdon tube to the atmosphere. The range and graduations of the pressure gage shall be governed by the vapor pressure of the sample being tested, in accordance with Table A1.1. Only accurate gages shall be continued in use. The calibration correction shall not be greater than 0.15 psi (0.3 kPa) for a 0 to 15-psi (0 to 30-kPa) gage or 0.3 psi (0.9 kPa) for a 0 to 30-psi (0 to 90-kPa) gage.

A1.3 Water Cooling Bath—A water cooling bath shall be provided of such dimensions that the sample containers and gasoline chambers may be completely immersed. Means for maintaining the bath at a temperature of 32 to 40 °F (0 to 4.5 °C) shall be provided.

NOTE A1.4. Solid carbon dioxide shall not be used to cool samples in storage or in the preparation of the air saturation step. Carbon dioxide is appreciably soluble in gasoline, and its use has been found to be the cause of erroneous vapor pressure data.

A1.4 Water Bath—The water bath shall be of such dimensions that the vapor pressure apparatus may be immersed to at least 1 inch (25 mm.) above the top of the air chamber. Means for maintaining the bath at a constant temperature of 100 ± 0.2 °F (37.8 ± 0.1 °C) shall be provided. In order to check this temperature, the bath thermometer shall be immersed to the 98 °F (37 °C) mark throughout the vapor pressure determination.

A1.5 Thermometers:

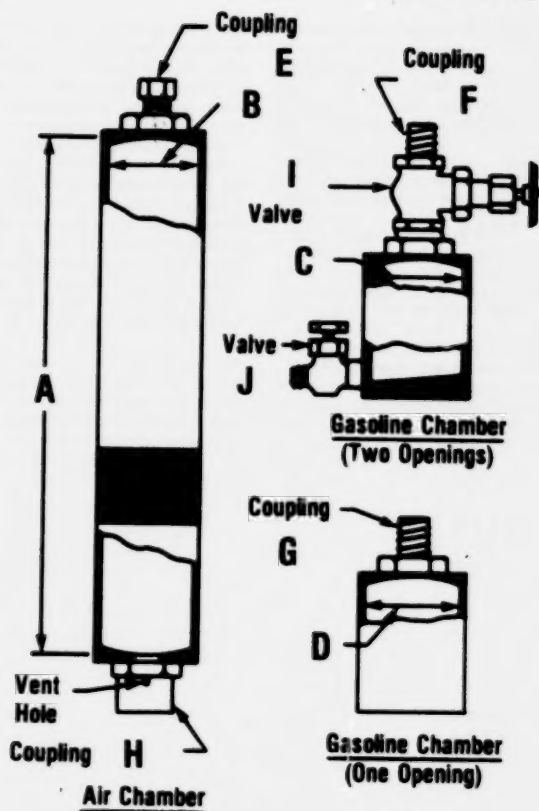
A1.5.1 For 100 °F (37.8 °C) Air Chamber Procedure—An ASTM Reid Vapor Pressure Thermometer 18F (18C) having a range from 94 to 108 °F (34 to 42 °C).

A1.5.2 For Water Bath—Use the ASTM Thermometer 18F (18C) described in A1.5.1.

A1.6 Mercury Manometer—A mercury manometer, having a range suitable for checking the pressure gage employed, shall be used. The manometer scale may be graduated in steps of 1 mm., 0.1 inch, 0.1 psi, or 0.001 bar.

TABLE A1.1—PRESSURE GAGE RANGE AND GRADUATION

Read vapor pressure		Scale range		Maximum numbered intervals		Maximum intermediate graduations	
psi	kPa	psi	kPa	psi	kPa	psi	kPa
4 and under	27.5 and under	0 to 5	0 to 35	1	5.0	0.1	0.5
5 to 12	30.0 to 75.0	0 to 15	0 to 100	3	15.0	0.1	0.5
13 to 26	90.0 to 180.0	0 to 30	0 to 200	5	25.0	0.2	1.0
27 to 36	70.0 to 250.0	0 to 45	0 to 300	5	25.0	0.2	1.0



DIMENSIONS OF VAPOR PRESSURE BOMB

Key	Description	In.
A	Air chamber, length	$10 \pm \frac{1}{4}$
B, C, D	Air and gasoline chambers, ID	$2 \pm \frac{1}{8}$
E	Coupling, ID min	$\frac{3}{16}$
F, G	Coupling, OD	$\frac{1}{4}$
H	Coupling, ID	$\frac{1}{2}$
I	Valve	$\frac{1}{2}$
J	Valve	$\frac{1}{4}$

Figure A1.1 Vapor Pressure Bomb

ANNEX A2. GASOLINE, GASOLINE-OXYGENATE BLENDS, NAPHTHA, METHYL CYCLOPENTANE, CYCLOPENTANE, N-PENTANE, METHYL TERT-BUTYL ETHER, TERT-AMYL METHYL ETHER

- A2.1 Danger—Extremely flammable. Vapors harmful if inhaled. Vapors may cause flash fire.
- A2.2 Keep away from heat, sparks, and open flame.
- A2.3 Keep container closed.
- A2.4 Use with adequate ventilation.
- A2.5 Avoid build-up of vapors and eliminate all sources of ignition, especially nonexplosion-proof electrical apparatus and heaters.
- A2.6 Avoid prolonged breathing of vapor or spray mist.
- A2.7 Avoid prolonged or repeated skin contact.

ANNEX A3. ACETONE

- A3.1 Danger—Extremely flammable. Vapors may cause flash fire.
- A3.2 Keep away from heat, sparks, and open flame.
- A3.3 Keep container closed.
- A3.4 Use with adequate ventilation.
- A3.5 Avoid build-up of vapors, and eliminate all sources of ignition, especially nonexplosion-proof electrical apparatus and heaters.
- A3.6 Avoid prolonged breathing of vapor or spray mist.
- A3.7 Avoid contact with eyes or skin.

METHOD 2—HERZOG SEMI-AUTOMATIC METHOD

1. Scope.

1.1 This test method covers the determination of the absolute vapor pressure of gasolines and gasoline-oxygenate blends using the Herzog Semi-Automatic Apparatus.¹ Test procedures will follow Method 1 except for the additions and changes as noted.

2. Summary of method.

2.1 The chilled liquid chamber is filled with a chilled sample and connected to the heated air chamber by means of a screwed connection. The assembled test chambers are immersed in a constant temperature bath controlled to $100 \pm 0.2^\circ \text{F}$ ($37.8 \pm 0.1^\circ \text{C}$) and rotated systematically until a constant pressure is observed on the pressure gauge (approximately 10 minutes). The pressure observed, suitably corrected, is reported as the Reid vapor pressure.

3. Significance and use.

3.1 This method is to be used as an alternative to Method 1. The procedures are es-

entially the same except that they are modified to represent the use of the Herzog Semi-Automatic Apparatus. As is the case with Method 1, this is considered to be a "dry" method since it can be used to evaluate both gasoline and gasoline-oxygenate blends.

4. Apparatus.

4.1 The Herzog Semi-Automatic Apparatus is composed of air and liquid test chambers, a constant temperature bath and means for observing the absolute pressure developed in the test chamber during the test. The analyzer is designed to allow the testing of three samples simultaneously.

4.2 The Herzog Semi-Automatic Apparatus consists of the equipment and accessories listed

1	Waterbath, stainless steel, with motor-driven support bearings for the rotation of three sample test chamber assemblies simultaneously.
1	Electronic bath control unit with LED indicator providing temperature control of $\pm 0.1^\circ \text{F}$ (0.05°C) or better and maximum temperature cutoff and minimum liquid level protection.
1	Control thermometer ($35\text{--}40^\circ \text{C}$) and silicone rubber stopper.
1	Bath cover, stainless steel.
3	Liquid sample chambers.
3	Air chambers.
3	Special screw fittings with teflon spiral.
3	Pressure gauges.
1	Liquid chamber filling device.
1	Table key.
1	115V to 220W step-up transformer (if ordered for 115V operation).

5. Physical size and weight.

5.1 Net weight without accessories (empty): 16 pounds (35 kg).

5.2 Dimensions: $39 \times 20 \times 16.5$ inches ($86 \times 51 \times 42$ cm.).

6. Installation requirements.

6.1 Laboratory bench or table providing a work space approximately four feet wide by two feet deep.

6.2 One 220 or 115V 50/60 Hz, 1000 watt grounded receptacle.

6.3 Means for cooling the test sample and the liquid sample test chamber to $32\text{--}34^\circ \text{F}$ ($0\text{--}1^\circ \text{C}$).

7. Installation instructions.

The recommended installation procedure is outlined

7.1 Verify that the working voltage corresponds to the requirements of the analyzer.

¹ Manufactured by Walter Herzog, GMBH, D-6970, Lauda, West Germany.

7.2 Place and level the analyzer on a stable table or laboratory work bench near the required power supply.

7.3 Release all of the function keys on the control unit.

7.4 Fill the heating bath with water to the upper line on the guide tube for the bath control thermometer at the rear right of the bath.

7.5 Insert the bath control thermometer through the bored silicone rubber stopper (supplied) and place in the thermometer guide tube. (Be sure to coat the glass thermometer with a lubricant and wear puncture-proof gloves and safety glasses to avoid breakage and possible injury.)

7.6 Connect the analyzer to the power supply.

7.7 Press the "MAINS" key.

7.8 Press the "STIRRER" key; bath circulation will start.

7.9 Press the "HEATING" key; bath heater will start.

NOTE 1: It may be necessary to press the "START TROUBLE" switch to begin operation.

7.10 After the preset temperature is reached, the bath is regulated electronically. The bath's temperature stability is indicated by a string of LED's.

7.11 When the LED marked "0" lights, the bath temperature of 100 °F (37.8 °C) (factory set) has been reached.

NOTE 2: The LED indicator is an optical aid, indicating a deviation from a preset temperature. Compare the bath control thermometer with the LED indicator. Checking the temperature with a calibrated thermometer is recommended. If the bath temperature does not agree with the desired temperature, adjust as follows:

Above the string of LED's is an opening marked "TEMP", behind which is a potentiometer for adjusting the bath temperature. The bath temperature can be raised, using a screw driver, by turning the potentiometer clockwise and can be lowered by turning the potentiometer counterclockwise. The readjusted temperature is reached when the LED at the "0" mark lights up.

NOTE 3: Maximum bath temperature and level is provided. If the bath temperature should rise 4 °F (2 °C) above the set test temperature, or the water level should drop below the minimum acceptable level, heating and stirring will automatically shut off. After the problem is corrected, the heating and stirring can be reactivated by pressing the "START TROUBLE" key.

7.12 Remove the shipping screw from the back of the Bourdon precision pressure gauge and replace the screw with the screw that will be found in the small envelope taped to the front of the gauge.

7.13 Fasten the three pressure gauges to the appropriate vapor line connections

along the back of the analyzer with the union nut. *Make certain that the teflon seals are in place and the connection is vapor tight.*

8. Test procedure.

Observe all sections of Method 1 from section 5, "Reagents," through section 11, "Precision and accuracy," except for the following changes:

7.4 Preparation of Air Chamber—Observe the apparatus preparation procedure of section 8.5. Stopper the lower connection of the Herzog air chamber with a #3 rubber stopper and the vent hole with either a #000 cork or a small rubber stopper. The lower connection of the Herzog air chamber may also be stoppered by utilizing a cap threaded to match the threads of the air chamber or by attaching a spare fuel chamber to the air chamber. In any procedure used, the interior surfaces of the vapor pressure apparatus and the sample must be kept completely free of water. Connect the spiral tubing T handle coupling to the air chamber and the quick action coupling to the gage or transducer connection. Immerse the air chamber in the water bath maintained at 100 °F ± 0.2 °F (37.8 °C ± 0.1 °C) for not less than 10 minutes just prior to coupling it with the gasoline chamber. Do not remove the air chamber from the bath until the gasoline chamber has been filled with sample as described in 8.1.

8.3 Introduction of the apparatus into bath. Tilt the assembled apparatus to 20° to 30° downward for four or five seconds to allow the sample to flow into the vapor chamber without getting into the tube extending into the vapor chamber. Place the assembled apparatus in the water bath (maintained at 100 °F ± 0.2 °F (37.8 °C ± 0.1 °C)) in such a way that the base of the gasoline chamber engages the drive coupling and the other end of the assembly rests on its support bearing. Observe the apparatus for leakage throughout the test. Discard the test at any time a leak is detected.

8.4 Measurement of vapor pressure. After the assembled vapor pressure apparatus has been immersed in the bath for at least 5 minutes, tap the pressure gage lightly and observe the reading. Repeat the tapping at intervals of not less than 2 minutes until two consecutive readings agree. (Tapping is not necessary with the transducer model but reading intervals should be the same.) Record this value as the "uncorrected vapor pressure". Refer to the gage on transducer calibration for the respective unit and add or subtract from the observed uncorrected value any offset indicated by the calibration in that range. Record this value as the Reid vapor pressure of the sample.

8.5 Preparation of apparatus for next test. Either of the cleaning procedures listed in

sections 8.5.1 and 8.5.2 may be used to prepare the apparatus for the next test.

8.5.1 Disconnect the quick action and T handle couplings. Separate the air and gasoline chambers and discard the contained sample. Thoroughly purge the air chamber of residual sample by filling it with warm water above 90 degrees F (32 degrees C) and allowing it to drain (Note 7). Repeat this purging at least five times. Rinse both chambers and sample transfer connection several times with hot water, then several times with acetone, then dry by blowing dried and filtered compressed air or pulling a vacuum. Assure that no liquid is present in the T handle fitting or spiral tubing by pulling a vacuum through the tubing. Stopper the gasoline chamber and place it in the refrigerator or ice-water bath for the next test.

8.5.2 Disconnect the quick action and T handle couplings. Separate the air and gasoline chambers and discard the contained sample. Rinse the chambers and the transfer connections in a manner such that all the inner surfaces are wetted with commercial grade n-pentane and drain into an appropriate container. Dry the interior surfaces with dried and filtered compressed air or by pulling a vacuum through them. Assure that no liquid is present in the T handle fitting or spiral tubing by pulling a vacuum or blowing dried and filtered compressed air through it. Stopper the gasoline chamber and place it in the refrigerator or ice-water bath for the next test.

[54 FR 11897, Mar. 22, 1989; 54 FR 27018, June 27, 1989, as amended at 55 FR 25836, June 25, 1990]

EFFECTIVE DATE NOTES: 1. At 55 FR 25835, June 25, 1990, Part 80, Appendix E, Method 1 was amended by revising note 4 to section 8.3, section 8.5 and note 7, and by adding new sections 8.5.1 and 8.5.2, effective August 24, 1990.

2. At 55 FR 25836, June 25, 1990, Part 80, Appendix E, Method 2 was amended by revising sections 7.4 and 8.5 and by adding new sections 8.5.1 and 8.5.2, effective August 24, 1990. For the convenience of the user, the superseded text appears as follows.

APPENDIX E—TESTS FOR DETERMINING REID VAPOR PRESSURE (RVP) OF GASOLINE AND GASOLINE-OXYGENATE BLENDS

METHOD 1—DRY RVP MEASUREMENT METHOD

8. . . .

NOTE 4: After the apparatus has been immersed in the bath, check the remaining sample for phase separation. If the sample is contained in a glass container, this observation can be made prior to sample transfer

(8.1). If the sample is contained in a non-transparent container, shake the sample vigorously for 5 seconds and then immediately pour a portion of the remaining sample into a clear glass container. Immediately after shaking this sample again for 5 seconds, observe the sample for phase separation. If this sample is not clear and bright, and free of a second phase, discard the test and the sample.

8.4 . . .

8.5 Preparation of apparatus for next test. Thoroughly purge the air chamber of residual sample by filling it with warm water above 90 °F (32 °C) and allowing it to drain (Note 7). Repeat this purging at least five times. After disconnecting the pressure gage from its manifold connection with the manometer, remove trapped fluid in the Bourdon tube of the gage by repeated centrifugal thrusts. This may be accomplished in the following manner: hold the gage between the palms of the hands with the right hand on the face side and the threaded connection of the gage forward. Extend the arms forward and upward at an angle of 45° with the coupling of the gage pointing in the same direction. Swing the arms downward through an arc of about 135° so that the centrifugal force aids gravity in removing the trapped liquid. Repeat this operation three times to expel all liquid. Purge the pressure gage by directing a small jet of air into its Bourdon tube for at least 5 minutes. Rinse both chambers and the sample transfer connection several times with hot water, then several times with acetone, then dry by blowing dried air or pulling a vacuum. Stopper the fuel chamber and place it in the refrigerator or ice-water bath for the next test.

NOTE 7: If the purging of the air chamber is done in a bath, be sure to avoid small and unnoticeable films of floating sample by keeping the bottom and top openings of the chamber closed as they pass through the water surface.

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METHOD 2—HERZOG SEMI-AUTOMATIC METHOD

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7. . . .

7.4 Fill the heating bath with distilled water to the upper line on the guide tube for the bath control thermometer at the rear right of the bath. (Water containing dissolved salts may shorten the life of the analyzer.)

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8. . . .

8.5 Preparation of apparatus for next test. Disconnect the quick action and T handle couplings. Separate the air and gasoline chambers and discard the contained sample. Thoroughly purge the air chamber of residual sample by filling it with warm water above 90 °F (32 °C) and allowing it to drain (Note 7). Repeat this purging at least five times. Rinse both chambers and sample transfer connection several times with hot water, then several times with acetone, then dry by blowing dried air or pulling a vacuum. Assure that no liquid is present in the T handle fitting or spiral tubing by pulling a vacuum through the tubing. Stopper the gasoline chamber and place it in the refrigerator or icewater bath for the next test.

APPENDIX F—TEST FOR DETERMINING THE QUANTITY OF ALCOHOL IN GASOLINE

METHOD 1—WATER EXTRACTION METHOD

1. Scope.

This test method covers the determination of the type and amount of alcohols in gasoline.

2. Summary of method.

Gasoline samples are extracted with water prior to analysis on a gas chromatograph (GC). The extraction eliminates hydrocarbon interference during chromatography. A known quantity of isopropanol is added to the fuel prior to extraction to act as an internal standard.

3. Sample description.

3.1 Sample in accordance with 40 CFR Part 80, Appendix D.

3.2 At least 100 ml. of gasoline suspected of containing ethanol and/or methanol are required.

4. Apparatus.

4.1 Gas chromatograph—A gas chromatograph equipped with a flame ionization detector.

4.2 Column—A gas chromatograph column, glass, 1800 by 6.35 cm. outside diameter, packed with chromosorb 102.

4.3 Recorder—A 1-mv recorder with a 1 second full scale response and a chart speed of 10 mm. per minute (0.4 inches per minute).

4.4 Syringe (100 μ l.) for adding the internal standard.

4.5 Pipet.

4.6 Injection syringe (10 μ l.).

4.7 Extraction syringe (1-5 ml.) with 3-inch stroke.

4.8 250 ml. ($\frac{1}{2}$ pint) glass sample bottles with screw caps or equivalent.

4.9 Calibration standard solutions extracted from gasoline containing known quantities of alcohols.

4.10 Reference standard solutions extracted from gasoline containing known quantities of alcohols.

4.11 Distilled water.

4.12 Reagent grade isopropanol.

4.13 Rubber gloves.

4.14 I.D. tags.

5. Precautions.

NOTE 1: Gasoline and alcohols are extremely flammable and may be toxic over prolonged exposure. Methanol is particularly hazardous. Persons performing this procedure must be familiar with the chemicals involved and all precautions applicable to each.

5.1 Extractions and dilutions must be performed in well-ventilated areas, preferably under a fume hood, away from open flames and sparks.

5.2 Rubber gloves must be worn during the handling of gasoline and alcohols.

5.3 Avoid breathing fumes from gasoline and alcohols, particularly methanol.

5.4 Gas cylinders must be properly secured and the hydrogen FID fuel must be segregated from the compressed air (oxidizer) tank.

6. Visual inspection.

6.1 Ensure that the samples do not contain sediment or separated phases prior to extraction.

6.2 Ensure adequate quantities of GC supply gases to maintain a run.

7. Test article preparation.

7.1 Gas chromatography—Use carrier gas, flow rates, detector and injection temperatures and column as specified in the GC manufacturer's specifications.

7.2 Sample extraction, preparation and analysis.

7.2.1 Label two 6 ml. vials with the sample identification number supplied with the original sample. The estimated percent alcohol from any screening tests must also be included on the label.

7.2.2 Pipet 4 ml. $\pm$ 0.01 ml. of sample into one of the vials. Label as vial #1.

7.2.3 Measure 100 μ l. (0.1 ml.) $\pm$ 0.5 μ l. of isopropanol into vial #1.

NOTE: This adds an internal standard to the sample which is required for accurate analysis.

7.2.4 Add 1 ml. $\pm$ 0.2 ml. of distilled water to the gasoline sample in vial #1 and shake for 10 seconds.

7.2.5 Allow the mixture to separate into two phases (at least 5 minutes).

7.2.6 Carefully draw off the aqueous (lower) phase using a 5 ml. syringe and long needle.

NOTE: Be careful not to allow any of the gasoline phase to get into the needle. Leave a small amount (approximately 0.2 ml.) of the aqueous phase in the vial.

7.2.7 Transfer the aqueous phase into the other 6 ml. vial (vial #2).

7.2.8 Repeat steps 7.2.4 to 7.2.6 two more times.

7.2.9 Fill vial #2 (the aqueous phase) to 4 ml. $\pm$ 0.05 ml. with distilled water.

7.2.10 Retain the remaining original gasoline sample (not the gasoline phase).

7.2.11 Discard the extracted gasoline phase in vial #1 in an appropriate manner.

7.2.12 Perform a second extraction on one sample in every 20. This sample is to be labeled with the sample number and as a duplicate and run as a normal sample.

7.2.13 Transfer approximately 2 ml. of the aqueous solution to vials compatible with the autosampler. Tag the vial with the sample number.

7.2.14 Perform analysis of the sample according to the GC manufacturer's specifications.

7.3 Standards.

7.3.1 Calibration standard solutions (made in gasoline).

7.3.1.1 Reagent grade or better alcohols (including undenatured ethanol) are to be diluted with regular unleaded gasoline. The isopropanol internal standard is to be added during extraction of the alcohols. Newly acquired stocks of reagent grade alcohols shall be diluted to 10% with hydrocarbon-free water and analyzed for contamination by GC before use.

7.3.1.2 Required calibration standards (%) by volume in gasoline):

Alcohol	Range (percent)	Standard (MIN)
Methanol	0.5-12	5
Ethanol	0.5-11	5

The standards should be as equally spaced within the range as possible and may contain more than one alcohol.

NOTE: Level #1 must contain all of the alcohols.

8. Quality control provisions.

8.1 Alcohol(s) in water solution may be used to characterize the GC. The resulting

characterization always reflects the absolute sensitivity of the instrument to each alcohol.

8.2 Calibration standards are made by extraction of known alcohol(s) in gasoline blends. These standards account for inaccuracies caused by incomplete extraction of alcohols.

8.3 The addition of isopropanol as an internal standard reduces errors caused by variations in injection volumes, and further reduces inaccuracies caused by incomplete extraction of alcohols.

8.4 Sufficient sample should be retained to permit reanalysis.

8.5 Running averages of reference standards data must not exceed 0.75% of applicable limits or investigation should be started for the cause of such variation.

9. Calculations.

9.1 Calculate purity of component as follows:

$$P_i = \frac{A_i}{\Sigma A} \quad \text{expressed as a decimal fraction, that is 0.999}$$

where:

P_i = purity of component i,

A_i = area of response of component i, and

ΣA = total area response of all components.

9.2 Calculate response factors as follows:

$$F_i = \frac{A_u \times W_i \times P_i}{A_i \times W_u \times P_u}$$

where:

F_i = response factor for component of interest i,

A_i = area response for component of interest i,

A_u = area response of internal standard,

W_i = weight of component of interest i (be sure to consider all sources),

W_u = weight of internal standard,

P_i = purity of component of interest i as determined in 9.1 expressed as a decimal, and

P_u = purity of internal standards as determined in 9.1 expressed as a decimal.

9.3 Calculate the percent alcohols as follows:

$$C_i = \frac{W_u \times A_i \times F_i}{W_i \times A_u} \times 100 = \quad \text{weight \% component i}$$

where:

A_i = peak area component i ,
 A_{is} = peak area of internal standard,
 W_i = weight of sample,
 W_{is} = weight of internal standard, and
 F_i = response factor for component i .

10. Report.

10.1 Report results to the nearest 0.1%.

11. Precision and accuracy.

11.1 Precision—The precision of this test method has not been determined.

11.2 Accuracy—The accuracy of this test method has not been determined.

METHOD 2—TEST METHOD FOR DETERMINATION OF C₁ TO C₄ ALCOHOLS AND MTBE IN GASOLINE BY GAS CHROMATOGRAPHY

1. Scope.

1.1 This test method covers a procedure for determination of methanol, ethanol, isopropanol, n-propanol, isobutanol, sec-butanol, tert-butanol, n-butanol, and methyl tertiary butyl ether (MTBE) in gasoline by gas chromatography.

1.2 Individual alcohols and MTBE are determined from 0.1 to 10 volume %. Any sample found to contain greater than 10 volume % of an alcohol or MTBE shall be diluted to concentrations within these limits.

1.3 SI (metric) units of measurement are preferred and used throughout this standard. Alternative units, in common usage, are also provided to improve the clarity and aid the user of this test method.

1.4 This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2. Referenced documents.

2.1 ASTM Standards:

D 4307 Practice for Preparation of Liquid Blends for Use as Analytical Standards¹

D 4626 Practice for Calculation of Gas Chromatographic Response Factors¹

E 260 Practice for Packed Column Gas Chromatographic Procedures²

E 355 Practice for Gas Chromatography Terms and Relationships²

2.2 EPA Regulations:

40 CFR Part 80 Appendix D

3. Descriptions of terms specific to this standard.

3.1 MTBE—methyl tertiary butyl ether.

¹ Annual Book of ASTM Standards, Vol. 05.03.

² Annual Book of ASTM Standards, Vol. 14.01.

3.2 Low volume connector—a special union for connecting two lengths of tubing 1.6 mm inside diameter and smaller. Sometimes this is referred to as a zero dead volume union.

3.3 Oxygenates—used to designate fuel blending components containing oxygen, either in the form of alcohol or ether.

3.4 Split ratio—a term used in gas chromatography using capillary columns. The split ratio is the ratio of the total flow of the carrier gas to the sample inlet versus the flow of carrier gas to the capillary column. Typical values range from 10:1 to 500:1 depending upon the amount of sample injected and the type of capillary column used.

3.5 WCOT—abbreviation for a type of capillary column used in gas chromatography that is wall-coated open tubular. This type of column is prepared by coating the inside of the capillary with a thin film of stationary phase.

3.6 TCEP—1,2,3, tris-2-cyanoethoxypropane—a gas chromatographic liquid phase.

4. Summary of test method.

4.1 An internal standard, tertiary amyl alcohol, is added to the sample which is then introduced into a gas chromatograph equipped with two columns and a column switching valve. The sample first passes onto a polar TCEP column which elutes lighter hydrocarbons to vent and retains the oxygenated and heavier hydrocarbons. After methylcyclopentane, but before MTBE elutes from the polar column, the valve is switched to backflush the oxygenates onto a WCOT non-polar column. The alcohols and MTBE elute from the non-polar column in boiling point order, before elution of any major hydrocarbon constituents. After benzene elutes from the non-polar column, the column switching valve is switched back to its original position to backflush the heavy hydrocarbons. The eluted components are detected by a flame ionization or thermal conductivity detector. The detector response, proportional to the component concentration, is recorded; the peak areas are measured; and the concentration of each component is calculated with reference to the internal standard.

5. Significance and use.

5.1 Alcohols and other oxygenates may be added to gasoline to increase the octane number. Type and concentration of various oxygenates are specified and regulated to ensure acceptable commercial gasoline quality. Drivability, vapor pressure, phase separation, and evaporative emissions are some of the concerns associated with oxygenated fuels.

5.2 This test method is applicable to both quality control in the production of gasoline and for the determination of deliberate or

extraneous oxygenate additions or contamination.

6. Apparatus.

6.1 Chromatograph:

6.1.1 A gas chromatographic instrument which can be operated at the conditions given in Table 1, and having a column

switching and backflushing system equivalent to Fig. 1. Carrier gas flow controllers shall be capable of precise control where the required flow rates are low (Table 1). Pressure control devices and gages shall be capable of precise control for the typical pressures required.

TABLE 1—CHROMATOGRAPHIC OPERATING CONDITIONS

Temperatures		Flows, mL/min		Other parameters: Carrier gas, helium	
Column oven, °C.....	60	To injector.....	75	Sample size, μ L.....	3
Injector, °C.....	200	Column.....	5	Split ratio.....	15:1
Detector—TCD, °C.....	200	Auxiliary.....	3	Backflush, min.....	0.2-0.3
FID, °C.....	250	Makeup.....	18	Valve reset time, min.....	8-10
Valve, °C.....	60			Total analysis time, min.....	18-20

6.1.2 Detector—A thermal conductivity detector or flame ionization detector may be used. The system shall have sufficient sensitivity and stability to obtain a recorded deflection of at least 2 mm at a signal-to-noise ratio of at least 5 to 1 for 0.005 volume % concentration of an oxygenate.

6.1.3 Switching and backflushing valve—A valve, to be located within the gas chromatographic column oven, capable of performing the functions described in Section 11, and illustrated in Fig. 1. The valve shall be of low volume design and not contribute significantly to chromatographic deterioration.

6.1.3.1 Valco Model No. CM-VSV-10-HT, 1.6-mm ($\frac{1}{16}$ -in.) fittings. This particular valve was used in the majority of the analyses used for the development of Section 15.

6.1.3.2 Valco Model No. C10W, 0.8-mm ($\frac{1}{8}$ -in.) fittings. This valve is recommended for use with columns of 0.32-mm inside diameter and smaller.

6.1.4 Although not mandatory, an automatic valve switching device is strongly recommended to ensure repeatable switching times. Such a device should be synchronized with injection and data collection times. If no such device is available, a stopwatch, started at the time of injection, should be used to indicate the proper valve switching time.

6.1.5 Injection system—The chromatograph should be equipped with a splitting-type inlet device. Split injection is necessary to maintain the actual chromatographed sample size within the limits of column and detector optimum efficiency and linearity.

6.1.6 Sample introduction—Any system capable of introducing a representative sample into the split inlet device. Microbore syringes, automatic syringe injectors, and liquid sampling valves have been used successfully.

6.2 Data presentation or calculation, or both:

6.2.1 Recorder—A recording potentiometer or equivalent with a full-scale deflection of 5 mV or less. Full-scale response time should be 1 s or less with sufficient sensitivity and stability to meet the requirements of 6.1.2.

6.2.2 Integrator or computer—Devices capable of meeting the requirements of 6.1.2, and providing graphic and digital presentation of the chromatographic data, are recommended for use. Means shall be provided for determining the detector response. Peak heights or areas can be measured by computer, electronic integration or manual techniques.

6.3 Columns, two as follows:

6.3.1 Polar column—This column performs a pre separation of the oxygenates from volatile hydrocarbons in the same boiling point range. The oxygenates and remaining hydrocarbons are backflushed onto the non-polar column in section 6.3.2. Any column with equivalent or better chromatographic efficiency and selectivity to that described in 6.3.1.1 can be used. The column shall perform at the same temperature as required for the column in 6.3.2.

6.3.1.1 TCEP micro-packed column, 560 mm (22 in.) by 1.6-mm ($\frac{1}{16}$ -in.) outside diameter by 0.38-mm (0.015-in.) inside diameter stainless steel tube packed with 0.14 to 0.15g of 20% (mass/mass) TCEP on 80/100 mesh Chromosorb P(AW). This column was used in the (ASTM) cooperative study to provide the Precision and Bias data referred to in Section 15.

6.3.2 Non-polar (analytical) column—Any column with equivalent or better chromatographic efficiency and selectivity to that described in 6.3.2.1 and illustrated in Fig. 2 can be used.

6.3.2.1 WCOT methyl silicone column, 30m (1181 in.) long by 0.53 mm (0.021-in.) inside diameter fused silica WCOT column with a 2.6- μ m film thickness of cross-linked methyl siloxane. This column was used in

the (ASTM) cooperative study to provide the Precision and Bias data referred to in Section 15.

7. Reagents and materials.

7.1 Carrier gas—Carrier gas appropriate to the type of detector used. Helium has been used successfully. The minimum purity of the carrier gas used must be 99.95 mol %.

7.2 Standards for calibration and identification—Standards of all components to be analyzed and the internal standard are required for establishing identification by retention as well as calibration for quantitative measurements. These materials shall be of known purity and free of the other components to be analyzed.

NOTE 1.—Warning—These materials are flammable and may be harmful or fatal if ingested or inhaled.

7.3 Preparation of calibration blends—For best results, these components must be added to a stock gasoline or petroleum naphtha, free of oxygenates (Warning—See Note 2). Refer to Test Method D 4307 for preparation of liquid blends. The preparation of several different blends, at different concentration levels covering the scope of the method, is recommended. These will be used to establish the linearity of the component response.

NOTE 2.—Warning—Extremely flammable. Vapors harmful if inhaled.

7.4 Methylene chloride—Used for column preparation. Reagent grade, free of non-volatile residue.

NOTE 3.—Warning—Harmful if inhaled. High concentrations may cause unconsciousness or death.

8. Preparation of column packings.

8.1 TCEP column packing:

8.1.1 Any satisfactory method, used in the practice of the art that will produce a column capable of retaining the C₁ to C₆ alcohols and MTBE from components of the same boiling point range in a gasoline sample. The following procedure has been used successfully.

8.1.2 Completely dissolve 10 g of TCEP in 100 mL of methylene chloride. Next add 40 g of 80/100 mesh Chromosorb P(AW) to the TCEP solution. Quickly transfer this mixture to a drying dish, in a fume hood, without scraping any of the residual packing from the sides of the container. Constantly, but gently, stir the packing until all of the solvent has evaporated. This column packing can be used immediately to prepare the TCEP column.

9. Preparation of micro-packed TCEP column.

9.1 Wash a straight 560 mm length of 1.6-mm outside diameter (0.38-mm inside di-

ameter) stainless steel tubing with methanol and dry with compressed nitrogen.

9.2 Insert 6 to 12 strands of silvered wire, a small mesh screen or stainless steel frit inside one end of the tube. Slowly add 0.14 to 0.15 g of packing material to the column and gently vibrate to settle the packing inside the column. When strands of wire are used to retain the packing material inside the column, leave 6.0 mm (0.25 in.) of space at the top of the column.

9.3 Column conditioning—Both the TCEP and WCOT columns are to be briefly conditioned before use. Connect the columns to the valve (see 11.1) in the chromatographic oven. Adjust the carrier gas flows as in 11.3 and place the valve in the RESET position. After several minutes, increase the column oven temperature to 120 °C and maintain these conditions for 5 to 10 min. Cool the columns below 60 °C before shutting off the carrier flow.

10. Sampling.

10.1 Gasoline samples to be analyzed by this test method shall be sampled in accordance with 40 CFR Part 80, Appendix D.

11. Preparation of apparatus and establishment of conditions.

11.1 Assembly—Connect the WCOT column to the valve system using low volume connectors and narrow bore tubing. It is important to minimize the volume of the chromatographic system that comes in contact with the sample, otherwise peak broadening will occur.

11.2 Adjust the operating conditions to those listed in Table 1, but do not turn on the detector circuits. Check the system for leaks before proceeding further.

11.3 Flow rate adjustment.

11.3.1 Attach a flow measuring device to the column vent with the valve in the RESET position and adjust the pressure to the injection port to give 5.0 mL/min flow (14 psig). Soap bubble flow meters are suitable.

11.3.2 Attach a flow measuring device to the split injector vent and adjust flow from the split vent using the A flow controller to give a flow of 70 mL/min. Recheck the column vent flow set in 11.3.1 and adjust if necessary.

11.3.3 Switch the valve to the BACK-FLUSH position and adjust the variable restrictor to give the same column vent flow set in 11.3.1. This is necessary to minimize flow changes when the valve is switched.

11.3.4 Switch the valve to the inject position RESET and adjust the B flow controller to give a flow of 3.0 to 3.2 mL/min at the detector exit. When required for the particular instrumentation used, add makeup flow or TCD switching flow to give a total of 21 mL/min at the detector exit.

11.4 When a thermal conductivity detector is used, turn on the filament current and

allow the detector to equilibrate. When a flame ionization detector is used, set the hydrogen and air flows and ignite the flame.

11.5 Determine the Time of Backflush—The time to backflush will vary slightly for each column system and must be determined experimentally as follows. The start time of the integrator and valve timer must be synchronized with the injection to accurately reproduce the backflush time.

11.5.1 Initially assume a valve BACKFLUSH time of 0.23 min. With the valve RESET, inject 3 μ L of a blend containing at least 0.5% or greater oxygenates (7.3), and simultaneously begin timing the analysis. At 0.23 min., rotate the valve to the BACKFLUSH position and leave it there until the complete elution of benzene is realized. Note this time as the RESET time, which is the time at which the valve is returned to the RESET position. When all of the remaining hydrocarbons are backflushed the signal will return to a stable baseline and the system is ready for another analysis. The chromatogram should appear similar to that illustrated in Fig. 2.

11.5.2 It is necessary to optimize the valve BACKFLUSH time by analyzing a standard blend containing oxygenates. The correct BACKFLUSH time is determined experimentally by using valve switching times between 0.2 and 0.3 min. When the valve is switched too soon, C_3 and lighter hydrocarbons are backflushed and are co-eluted in the C₁ alcohol section of the chromatogram. When the valve BACKFLUSH is switched too late, part or all of the MTBE component is vented resulting in an incorrect MTBE measurement. Chromatograms resulting from incorrect valve times are shown in Figs. 3 and 4.

12. Calibration and standardization.

12.1 Identification—Determine the retention time of each component by injecting small amounts either separately or in known mixtures or by comparing the relative retention times with those in Table 2.

12.2 Standardization—The area under each peak in the chromatogram is considered a quantitative measure of the corresponding compound. Measure the peak area of each oxygenate and of the internal standard by either manual methods or electronic integrator. Calculate the relative volume response factor of each oxygenate, relative to the internal standard, according to Test Method D 4626.

TABLE 2—RETENTION CHARACTERISTICS FOR TCEP/WCOT COLUMN SET CONDITIONS AS IN TABLE 1

Component	Retention time, min	Relative retention time (t-amylicol = 1.00)
Methanol.....	3.21	0.44
Ethanol.....	3.58	0.50
Isopropanol.....	3.95	0.56
tert-Butanol.....	4.31	0.61
n-Propanol.....	4.75	0.68
MTBE.....	5.29	0.76
sec-Butanol.....	5.63	0.82
Isobutanol.....	6.33	0.93
n-Butanol.....	7.55	1.10
Benzene.....	7.88	1.17

13. Procedure.

13.1 Preparation of sample—Precisely add a quantity of the internal standard to an accurately measured quantity of sample. Concentrations of 1 to 5 volume percent have been used successfully.

13.2 Chromatographic analysis—Introduce a representative aliquot of the sample, containing internal standard, into the chromatograph using the same technique as used for the calibration analyses. An injection volume of 3 μ L with a 15:1 split ratio has been used successfully.

13.3 Interpretation of chromatogram—Compare the results of sample analyses to those of calibration analyses to determine identification of oxygenates present.

14. Calculation.

14.1 After identifying the various oxygenates, measure the area of each oxygenate peak and that of the internal standard. Calculate the volume percent of each oxygenate as follows:

$$V_i = \frac{V_s \times PA_i \times 100}{PA_s \times S_i \times V_G}$$

where:

V_i =volume percent of oxygenate to be determined,

V_s =volume of internal standard (tert-amyl alcohol) added,

V_o =volume of gasoline sample taken,

PA_i =peak area of the oxygenate to be determined,

PA_s =peak area of the internal standard (tert-amyl alcohol), and

S_i =relative volume response factor of each component (relative to the internal standard).

14.2 Report the volume of each oxygenate. If the volume percent exceeds 10%, dilute the sample to a concentration lower than 10% and repeat the procedures in sections 13 and 14.

15. Precision and bias.

15.1 Precision—The precision of this test method as determined by statistical examination of the interlaboratory test results is as follows:

15.1.1 Repeatability—The difference between successive results obtained by the same operator with the same apparatus under constant operating conditions on identical test materials would, in the long run, in the normal and correct operation of the test method exceed the following values only in one case in twenty (see Table 3).

Methanol (V + 0.070)	0.086	×	Isobutanol (V + 0.086)	0.084	×
Ethanol 0.083	×	(V + 0.000)	...	sec-Butanol 0.014	×
Isopropanol (V + 0.150)	0.052	×	tert-Butanol (V + 0.398)	0.052	×
n-Propanol (V + 0.026)	0.040	×	n-Butanol (V + 0.020)	0.043	×
MTBE 0.104 × (V + 0.028)					

where V is the mean volume percent.

15.1.2 Reproducibility—The difference between two single and independent results obtained by different operators working in different laboratories on identical material would, in the long run, exceed the following values only in one case in twenty (see Table 3).

Methanol (V + 0.070)	0.361	×	Isobutanol (V + 0.086)	0.179	×
Ethanol 0.373	×	(V + 0.000)	...	sec-Butanol 0.277	×
Isopropanol (V + 0.150)	0.214	×	tert-Butanol (V + 0.398)	0.178	×
n-Propanol (V + 0.026)	0.163	×	n-Butanol (V + 0.020)	0.415	×
MTBE 0.244 × (V + 0.028)					

where V is the mean volume percent.

15.2 Bias—Since there is no accepted reference material suitable for determining bias for the procedure in the test method, bias cannot be determined.

TABLE 3—PRECISION INTERVALS—DETERMINED FROM COOPERATIVE STUDY DATA SUMMARIZED IN SECTION 15

Components	Volume percent							
	0.20	0.50	1.00	2.00	3.00	4.00	5.00	6.00
Repeatability								
Methanol	0.02	0.05	0.09	0.18	0.26	0.35	0.44	0.51
Ethanol	0.02	0.04	0.08	0.17	0.25	0.33	0.42	0.50
Isopropanol	0.02	0.03	0.06	0.11	0.16	0.22	0.27	0.32
n-Propanol	0.01	0.02	0.04	0.08	0.12	0.16	0.20	0.24
tert-Butanol	0.03	0.05	0.07	0.12	0.18	0.23	0.28	0.33
sec-Butanol	0.01	0.01	0.01	0.02	0.02	0.03	0.03	0.03
Isobutanol	0.02	0.04	0.07	0.13	0.20	0.26	0.33	0.39
n-Butanol	0.01	0.02	0.04	0.09	0.13	0.17	0.22	0.28
MTBE	0.02	0.05	0.11	0.21	0.31	0.42	0.52	0.63
Reproducibility								
Methanol	0.10	0.21	0.39	0.75	1.11	1.47	1.83	2.19
Ethanol	0.07	0.19	0.37	0.75	1.12	1.49	1.87	2.24
Isopropanol	0.07	0.14	0.25	0.46	0.67	0.89	1.10	1.32
n-Propanol	0.04	0.09	0.17	0.33	0.49	0.66	0.82	0.98
tert-Butanol	0.10	0.16	0.25	0.43	0.60	0.78	0.96	1.14
sec-Butanol	0.12	0.20	0.28	0.39	0.48	0.55	0.62	0.68
Isobutanol	0.05	0.10	0.19	0.37	0.55	0.73	0.91	1.09
n-Butanol	0.09	0.22	0.42	0.84	1.25	1.67	2.08	2.50

TABLE 3—PRECISION INTERVALS—DETERMINED FROM COOPERATIVE STUDY DATA SUMMARIZED IN SECTION 15—Continued

Components	Volume percent							
	0.20	0.50	1.00	2.00	3.00	4.00	5.00	6.00
MTBE	0.05	0.12	0.23	0.45	0.68	0.90	1.13	1.35

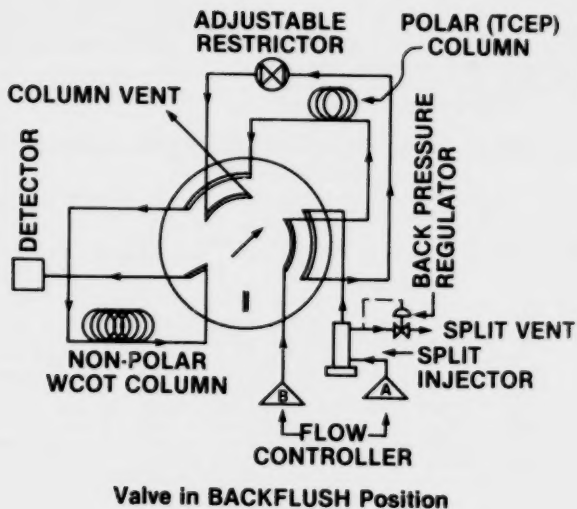
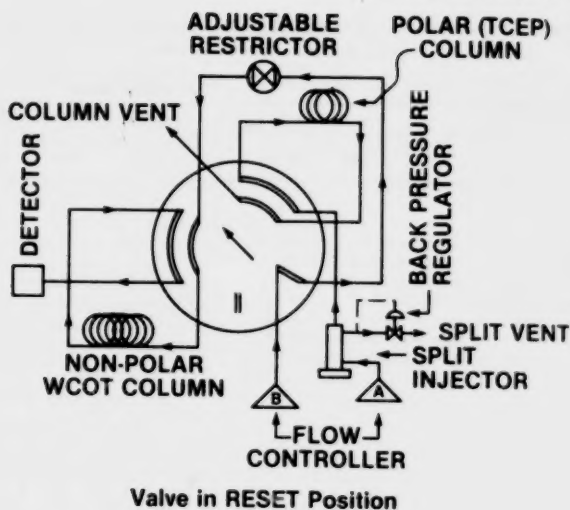


FIG. 1 Analysis of Oxygenates in Gasoline Schematic of Chromatographic System

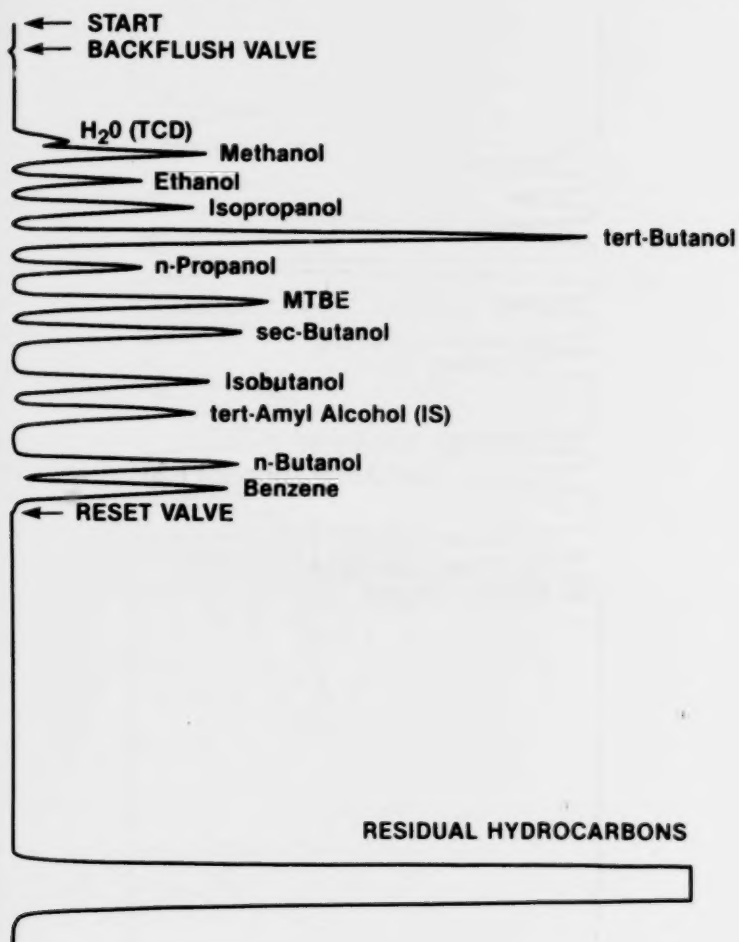


FIG. 2 Analysis of Oxygenates in Gasoline Example of Chromatographic Results

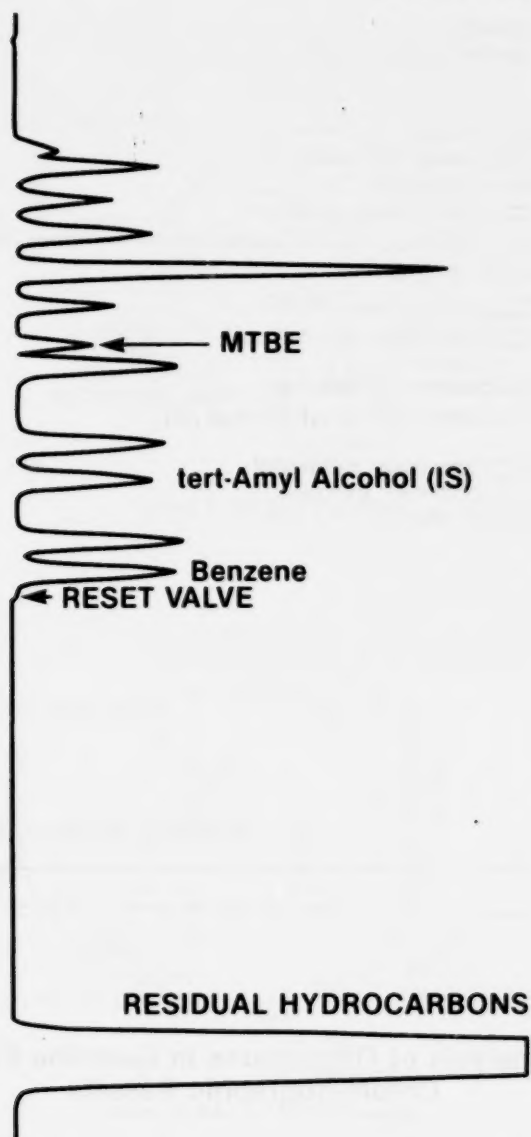


FIG. 3 Analysis of Oxygenates in Gasoline Example Chromatogram Showing Loss of MTBE Due to Venting with Light Hydrocarbons by Late Backflush Time

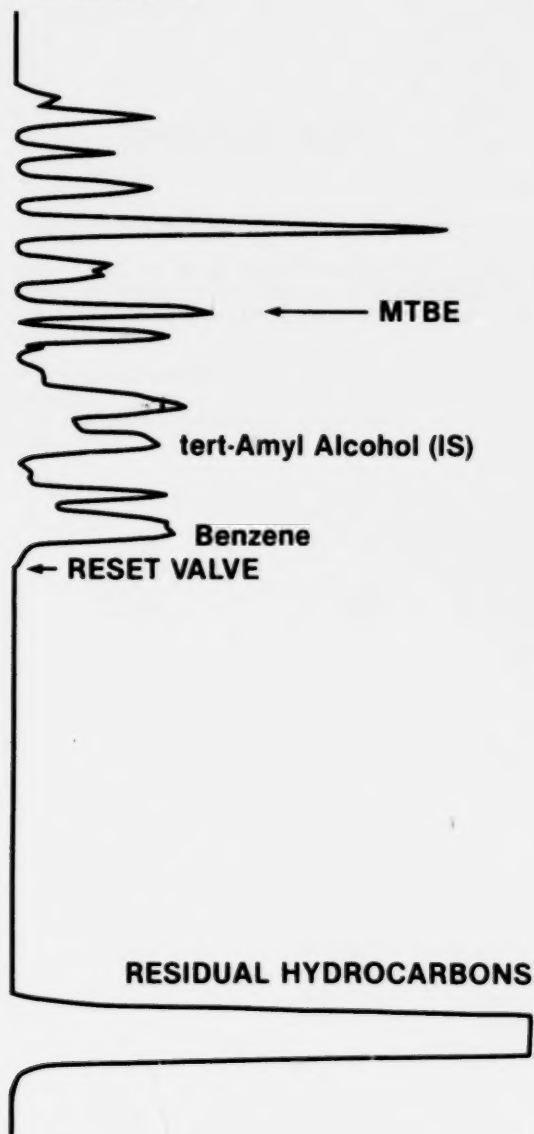


FIG. 4 Analysis of Oxygenates in Gasoline Example Chromatogram Showing Presence of Interferences Caused by Early Backflush Time

FINDING AIDS

A list of CFR titles, subtitles, chapters, subchapters and parts and an alphabetical list of agencies publishing in the CFR are included in the CFR Index and Finding Aids volume to the Code of Federal Regulations which is published separately and revised annually.

Material Approved for Incorporation by Reference
Table of CFR Titles and Chapters
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List of CFR Sections Affected

Material Approved for Incorporation by Reference

(Revised as of June 29, 1990)

The Director of the Federal Register has approved under 5 U.S.C. 552(a) and 1 CFR Part 51 the incorporation by reference of the following publications. This list contains only those incorporations by reference effective as of the revision date of this volume. Incorporations by reference found within a regulation are effective upon the effective date of that regulation. For more information on incorporation by reference, see the preliminary pages of this volume.

40 CFR, CHAPTER 1 (PARTS 61 TO 80): SUBCHAPTER C—AIR PROGRAMS ENVIRONMENTAL PROTECTION AGENCY

40 CFR

American National Standards Institute

1430 Broadway, New York, NY 10018

ANSI N13.1-1969 Guide to Sampling Airborne Radioactive Materials in Nuclear Facilities.

61.18(c);
61.93(b)(2)(ii);
61.107(b)(2)(ii);
Method 114,
paragraph 2.1 to
Appendix B of
Part 61

American Society for Testing and Materials

1916 Race Street, Philadelphia, Pennsylvania 19103

ASTM D439-81, Standard Specifications for Automotive Gasoline

80.2(d); 80.22(b)

ASTM D737-75, Standard Test Method for Air Permeability of Textile Fabrics.

61.23(a)

ASTM D 835-85, Standard Specification for Refined Benzene-485.....

61.18(a); 61.270(a)

ASTM D836-80, Standard Specification for Industrial Grade Benzene....

61.120(a)

ASTM D 836-84, Standard Specification for Industrial Grade Benzene ..

61.18(a); 61.270(a)

ASTM D2267-68 (Reapproved 1978), Aromatic in Light Naphtas and Aviation Gasolines by Gas Chromatography.

61.18(a)(4);
61.67(h)(1);
61.115(d)(1)

ASTM D 2359-85a, Standard Specification for Refined Benzene-535

61.18(a); 61.270(a)

ASTM D2382-76, Heat of Combustion of Hydrocarbon Fuse by Bomb Calorimeter (High Precision Method).

61.103(e)(3);
61.115(e)(3);
61.122(d)(3)

ASTM D2504-67 (Reapproved 1977) Noncondensable Gases in C3 and Lighter Hydrocarbon Products by Gas Chromatography.

61.103(b); 61.115(e);
61.122(d)

ASTM D2699-80, Standard Test Method for Knock Characteristics of Motor Fuels by the Research Method.

80.2(d)

ASTM D2700-81, Standard Test Method for Knock Characteristics of Motor and Aviation Fuels by the Motor Method.

80.2(d)

ASTM D2986-71 (Reapproved 1978), Standard Method for Evaluation of Air, Assay Media by the Monodisperse DOP (Diocetyl Phthalate) Smoke Test.

Appendix B to Part
61, Method 103,
par. 2.1.3; Method
104, par. 3.1.1;
61.18(a)(3)

ASTM D 4734-87 Standard Specification for Refined Benzene-545.....

61.18(a); 61.270(a)

Title 40—Protection of Environment

40 CFR, CHAPTER I (PARTS 61 TO 80): SUBCHAPTER C—AIR PROGRAMS—

Continued

ENVIRONMENTAL PROTECTION AGENCY—Continued

40 CFR

ASTM E 50-82 (Reapproved 1986), Standard Practice for Apparatus Reagents, and Safety Precautions for Chemical Analysis of Metals. 61.18(a)(7); Method 108C, par. 2.1.4, Appendix B to Part 61

Environmental Protection Agency

Copies available at: First International Bldg., 1201 Elm St., Dallas, TX 75270

Approved State Plans for designated facilities and pollutants 62.02(d)

Approved plan for Louisiana

Copies available at: Environmental Monitoring and Support Laboratory, Cincinnati, OH 45268

Method 601, Test Method for Purgeable Halocarbons, July 1982..... 61.67(g)(2)

U.S. Government Printing Office

Washington, DC 20402-9371; Telephone: 202-783-3238; Telefacsimile: 202-275-0019

Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, November 1986, as amended by Revision I, December 1987, order no. 955-001-00000-1; Method 8020, Aromatic Volatile Organics; Method 8021, Volatile Organic Compounds in Water by Purge and Trap Capillary Column Gas Chromatography with Photoionization and Electrolytic Conductivity Detectors in Series; Method 8240, Gas Chromatography/Mass Spectrometry for Volatile Organics; Method 8260, Gas Chromatography/Mass Spectrometry for Volatile Organics; Capillary Column Technique. 61.18(c); 61.355(c)(2)(iv)

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- V Agricultural Research Service, Department of Agriculture (Parts 500—599)
- VI Soil Conservation Service, Department of Agriculture (Parts 600—699)
- VII Agricultural Stabilization and Conservation Service (Agricultural Adjustment), Department of Agriculture (Parts 700—799)
- VIII Federal Grain Inspection Service, Department of Agriculture (Parts 800—899)
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